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VOL. 20. SEPTEMBER, 1934. NO. 131

JOURNAL OF THE INSTITUTION OF PETROLEUM TECHNOLOGISTS AND RECORD OF TRANSACTIONS.

FOUNDED 1913—INCORPORATED 1914.

PUBLISHED BY
THE INSTITUTION OF PETROLEUM TECHNOLOGISTS,
ALDINE HOUSE, BEDFORD STREET, STRAND, LONDON, W.C. 2.
AND PRINTED IN GREAT BRITAIN BY
W. SPRAIGHT AND SONS, LTD.,
98-99 FETTER LANE, LONDON, E.C. 4.

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THE INSTITUTION OF PETROLEUM TECHNOLOGISTS.

THE ONE HUNDRED AND FIFTY-EIGHTH GENERAL MEETING of the Institution of Petroleum Technologists was held at the House of the Royal Society of Arts, John Street, Adelphi, London, on Tuesday, May 8th, 1934, MR. T. DEWHURST, President, in the Chair.

The Secretary read the names of candidates nominated for election and the following list of members elected :—

As Member.—Henry Grafton Williams.

As Transfer to Member.—Michael Edmund Hubbard.

As Associate Members.—Ernest Hjalmar Ettlinger, Cyril William Martin, John Sydney Meredith, Aelwyn Williams-Gardner.

As Transfer to Associate Members.—Alfred George Gardegne de Chastelain, Harry Hyams, Hallowell Charles Hermann Thomas.

As Students.—Ian Stormont Bisset, Herbert Stephen Keernan.

The President said that the main business of the meeting was the reading of a paper by Harvey S. Garlick. Mr. Garlick was well known to members, as he had passed through every grade of membership, having been elected a Student in 1922, an Associate Member in 1926 and a Member in 1930. Members would recollect that Mr. Garlick in 1926 received the Students' Medal and Prize for an excellent paper on the Chemistry of Petroleum.

The following paper was then read :—

Lubricating Grease.

By

HARVEY S. GARLICK, F.I.C., B.Sc. (Hons. Lond.) (Member).

IN order to decide upon the most appropriate lubricant for any specific requirement, consideration must be given not only to the prevailing operating conditions but also to the adaptability of the selected product to the available means of application.

This involves a detailed knowledge of the physical characteristics of the lubricant if it is to function as intended.

Lubricating oils have received more attention and are more fully understood than greases. The reason is that there is not the possibility for so wide a variation in the different types of lubricating oils as there is in greases. Moreover, the physical characteristics such as viscosity, pour test and flash point by which lubricating oils are commonly classified have been standardized and rendered capable of checking in a ready and practical manner.

On the other hand, with greases the meaning and value of such properties as consistency, hardness, melting-point and colour are

frequently ambiguous to many, and are very often difficult to check or even define.

As a result, it is felt that the manufacture of greases, their properties and their function as lubricants are well worthy of discussion, for where to use grease or when to use oil is one of the prevailing problems in industrial plant lubrication to-day.

It is commonly reported that the first grease was manufactured shortly after the discovery of oil in Pennsylvania about seventy years ago. This was a simple type of axle grease made by mixing crude petroleum with rosin oil, then adding milk of lime and stirring until the mixture began to thicken. This may be so, but undoubtedly solid lubricants must have been known and used long before that time. Natural fats have always been to hand, and since soap-making is an ancient craft, it is inconceivable that the advantages of using a partially or wholly saponified product were not realised long before the days of petroleum products.

The technique and art of grease-making has not failed to keep pace with the rapid developments in industrial machinery. In the past a certain element of mystery has surrounded the subject of the manufacture of grease, and this has undoubtedly been fostered by the older type of grease-maker.

Admittedly grease-making is an art that has been developed to a high degree by such men, but too often their lack of technical training has led to their adopting certain practices that have no basis on chemical theory.

Increased speeds, bearing loads and pressures on gear teeth have given rise to conditions under which lubricating oil does not always afford the greatest measure of satisfaction. This in turn has led to a renewed study of lubricating greases. More and more chemists have become interested in the technical phases and closer co-operation with designers, manufacturers and users of mechanical equipment has brought about a marked improvement in the methods of grease manufacture and lubrication.

The result is that lubricating greases can no longer be regarded merely as an outlet for the by-products of the petroleum industry.

All raw materials entering into the composition of a grease are subjected to rigid specifications, and many of the most highly refined petroleum oils are used. Moreover, manufacturing processes are now more often than not controlled from the Laboratory.

The numerous products termed greases on the market to-day are so varied that it is almost impossible to give a definition of grease that will embrace all of them.

The most generally accepted description as proposed by the American Society of Testing Materials, regards grease as "a

combination of a petroleum product and a soap or a mixture of soaps, suitable for certain types of lubrication."

However, besides this type, we have admixtures of mineral oil and solid lubricants, and blends of residuum, waxes, uncombined fats, rosin oils and pitches, which, because of their solid or semi-solid nature, are also called greases.

The first group, comprising admixtures of oils with commonly termed solid lubricants such as graphite, mica, talc and sulphur, are rarely used except for the lubrication of an ill-fitting machine part, functioning under a heavy intermittent load, at slow speed. The filler or solid lubricant absorbs the force of impact, thereby preventing metal-to-metal contact, and at the same time tends to smooth any roughness of the bearing surfaces. Occasionally under the title of non-melting greases such products may be used for the lubrication of oven chains, mechanical stokers and similar appliances where the heat conditions are such as would cause charring of a soap-containing product.

The second group, consisting of various mixtures of viscous petroleum residuum and waxes, rosins, pitch and uncombined fats, possesses valuable characteristics for certain particular types of lubricating requirements, as for steel cables, chains, dredges, cranes and gears operating under water or exposed to the weather. Their high degree of metallic adhesion offers a protection against the operating conditions encountered.

Apart from just mentioning the above examples of pseudo-greases, it is the intention to confine this discussion to that classification of lubricating grease most conveniently defined as soap-thickened mineral oils.

How Greases are Made.

The ingredients used in the manufacture of lubricating greases include the saponifying agent or metallic base, the saponifiable fat and the mineral oil.

The manufacturing procedure, in general, is to heat the fat in contact with the metallic base until the saponification reaction is complete. The mineral oil is then slowly worked into the hot soap until a grease of the desired consistency is made.

There are some exceptions and variations to this general procedure which will be described later when discussing each type of grease.

By varying the raw materials and method of manufacture greases may be produced in many types, each having specific characteristics.

In turn, by varying the soap content but otherwise using the same materials, a range of greases may be manufactured having similar characteristics but varying in hardness.

When products of this nature are manufactured by the same process and are applied to the same general class of work, they are usually classified under what is known as a "grease series."

The type of saponifiable oil or fat is secondary in importance to the saponifying agent when considering the construction of a grease.

The more specific properties such as texture, degree of metallic adhesion and cohesive character of the grease body are controlled to a certain extent by selection of the fatty ingredient, but it is the saponifying agent or metallic base that determines the type of soap, thereby fixing the general physical properties of a grease and limiting the uses to which it can be put.

For this reason, it is most convenient to classify greases according to the kind of soap base used in their manufacture. Under such a classification we have :—

1. Lime Base Greases :
   (*a*) Cup Greases.
   (*b*) Sett Greases.
2. Soda Base Greases.
3. Lead Base Greases.
4. Aluminium Base Greases.
5. Miscellaneous types and special products.

1. Lime Base Greases.

Calcium or lime-soap greases are probably used to a greater extent than any other type of soap-thickened lubricant.

The commonly known varieties include the many grades of cup grease, pressure gun grease, ball and roller bearing grease, water-pump grease and axle grease. All types of mineral oils from a low viscosity spindle oil to a heavy residuum may be used in their manufacture.

Lime base greases may be sub-divided broadly as follows :—

(1) Cup Greases, the soap of which is made by the interaction of lime with the ordinary types of fats and fatty acids.

(2) Sett Greases, containing the lime soap of the acids present in rosin and rosin oil.

Lime Base Cup Greases. General Characteristics.—The various products known as cup greases constitute the most important class of grease lubricants, over half the production of the usual grease plant being of this type. They vary from a semi-fluid oil to a hard solid that will fracture when bent. In colour cup greases may vary from transparent pale yellow to jet black.

From a colloidal standpoint cup greases are emulsions of mineral lubricating oil and water, stabilised by lime soap. The external

phase is the mineral lubricating oil ; the water is the inner or dispersed phase.

Calcium oleate and calcium stearate are, from a practical consideration, insoluble in water, but have a slight solubility in mineral lubricating oil at normal temperatures.

On heating mineral lubricating oil and the calcium salts of the higher molecular weight fatty acids, the soaps will be dissolved, and on cooling a gel will be obtained. This mixture is unsatisfactory for lubrication purposes because of its tendency toward "sweating" oil and its instability to mechanical agitation. On stirring such a gel the soap is precipitated and the gel characteristics destroyed.

If water is introduced into the mixture of calcium soap and mineral lubricating oil at a temperature of 150–200° F., there can be obtained a stable product that is called a cup grease. The minimum quantity of water necessary to stabilise a cup grease is normally in the neighbourhood of 2–3 per cent. Cup greases rarely contain more than 5 per cent. of water. On the other hand, some fats are more soluble in particular types of mineral oils, and by suitable selection of both the fat or fats and the mineral oil, and by careful control of the manufacturing conditions, it is possible to produce a specialised type of cup grease containing well under 1 per cent. of water that is perfectly stable and free from separation of oil.

In view of what has been said, it will be appreciated that the ordinary cup greases are not applicable as lubricants where the temperature is liable to rise above 212° F. or even to remain high enough to cause evaporation of the inherent moisture. If in practice a cup grease loses its moisture, then separation of oil and soap results.

Excessive pressure may also cause separation of soap and oil, but for high-pressure applications the stability of a cup grease can be greatly improved by appropriate selection of the fatty ingredients. Incorporation of a high viscosity mineral oil is also beneficial in this respect.

The usual type of cup grease will liquefy at temperatures of 200 to 212° F., although by using certain specific fats this temperature can be raised considerably higher, especially in the stiffer greases of this type. Instability due to loss of moisture, however, will usually preclude their use at such temperatures.

The outstanding characteristics of lime soap greases are their insolubility in water and their short shear or buttery texture. Insolubility in water renders them ideal lubricants where it is desirable for the lubricant also to function as a seal to prevent ingress of moisture.

The texture of cup greases indicates a low degree of cohesion within the grease body, thereby reducing drag to the minimum and rendering them suitable for high-speed lubrication. The texture also offers high resistance to flow, rendering some sort of pressure system necessary for their general application. Because of this resistance to flow, the grease is not easily displaced by the centrifugal action of rotating parts, a definite advantage on bearings required to operate without attention for comparatively long periods of time.

Materials Used in Cup Grease Manufacture.

Soap Stock.—Cup greases may be prepared from any of the animal or vegetable oils or fats capable of being saponified by lime. The whole fats (triglycerides) or the fatty acids naturally occurring in them or derived from them may be employed.

Such factors as quality and viscosity of the mineral oil stock, colour, odour, texture and melting-point of the finished grease and the equipment available for the manufacturing process must be taken into consideration in the selection of the most appropriate soap stock for producing a particular grease.

Commercial fats of known origin are usually sufficiently uniform to insure satisfactory results. However, in order to maintain absolute uniformity in the products, close chemical control is necessary, as changes in colour, odour, melting-point, acidity or percentage of unsaponifiable matter will cause variation in quality in the finished product.

Purchasing specifications which include saponification number, acidity, melting-point, colour, odour and maximum tolerance for dirt and moisture should be issued for each stock in order to maintain the quality of shipments received. With less frequently used saponifiable oils, a number of special individual tests may be necessary to establish their identity.

The fats most commonly used in this country for the manufacture of the ordinary type of cup grease are the tallows, tallow oil and bone grease. Other fats less frequently used, or used for imparting some particular property to the resultant product, are lard oil, neatsfoot oil, palm oil, cottonseed oil, castor oil, rapeseed oil, fish oil, linseed oil and hydrogenated oils.

Many manufacturers use oleic acid in combination with fats in making cup greases, particularly when the reaction is carried out in an open kettle and speed of reaction is an important factor.

The use of stearic acid is often resorted to for the production of greases of high melting-point, but considerable care is necessary in its use in order to avoid the formation of hard lumps of calcium stearate.

A relatively new development is the manufacture of cup greases from wool grease stearines and oleines, tallow fatty acids, cotton-seed stearine and the mixed fatty acids obtained by splitting a fat or mixture of fats. These may be used alone or with one or more of the fats already mentioned.

Lime.—Formerly it was common practice for lump quick-lime to be used for cup grease manufacture. This was slaked, and was then made into a milk of lime and passed through wire gauze to remove impurities. By this procedure it was never possible to control the exact strength of the lime suspension; dirt and grit were impossible to eliminate, the amount of wastage was very high, and the resultant lime suspension was excessively diluted with water, which had to be driven off during the grease making, thus prolonging the time and increasing the expense of manufacture.

A much more satisfactory procedure is to use hydrated lime. It is now possible to purchase chemically pure hydrated lime of uniform calcium-oxide strength, entirely free from dirt and grit, and it is such material which forms the basis of most cup grease manufactured to-day. This lime is available in 56-lb. and 112-lb. paper bags, extremely easy to handle, and can be used either in its dry state or with just sufficient moisture to aid saponification.

Variation in the strength of the lime used can cause considerable variation in the consistency of the resulting cup greases so that it is always desirable to check the quality of fresh deliveries.

The presence of magnesium in the lime causes irregular results, and should be avoided. Silica should be under 0·5 per cent., and iron oxide and alumina should not exceed 0·2 per cent. in an hydrated lime to be used for manufacture of the highest quality greases.

A feature known but insufficiently investigated is the question of particle size and the reactivity of various hydrated limes. Some hydrated limes will react with fats, and particularly with fatty acids, far more quickly than others that may be analytically similar. Therefore careful selection must be made in order to obtain the best possible results.

The chemical analysis of a commercial hydrated lime suitable for grease manufacture is as follows :—

	CaO.	MgO.	Al_2O_3.	Fe_2O_3.	SiO_2.	SO_3.	CO_2.	H_2O.
I. ..	75·2	0·40	0·19	0·04	0·46	0·26	0·44	23·0
II. ..	98·2	0·52	0·25	0·05	0·60	0·34	—	—

I. Represents the true analysis.

II. Represents the analysis reduced to that of water-free and CO_2 free lime.

Mineral Oil.

The mineral oil content constitutes from 70 to 90 per cent. of the usual cup greases and is very important in that many of the

properties of the oil are imparted to the grease. The types of mineral oil employed at the present time vary within wide limits, but the oft-repeated statement " that the oil contained in a cup grease should conform in all respects with the oil that would be specified for similar operating conditions were it possible to use oil instead of grease " is by no means true. An ordinary cup grease made with a spindle oil will often effectively lubricate under conditions in which the oil by itself would be useless. The lime-soap not only acts as a pad or sponge to hold the oil in contact with the surfaces to be lubricated, but definitely contributes to the lubricating value of the oil.

Nevertheless the statement quoted above serves to bring out the truth that the best results can be obtained only from greases in which the oil is appropriately selected. Although viscosity is by no means a true index to lubricating value, it stands unquestioned that a heavy bodied oil possesses a stronger film than a light bodied oil. On the other hand, the heavier the body of the oil used in making a grease, the greater will be its drag and the more difficult it will be to feed it to bearings through pressure equipment.

The majority of ordinary cup greases are made with a light lubricating oil having a Redwood viscosity in the region of 200 secs. at 70° F., but a further grade of cup grease, having an oil of 300 to 350 secs. is manufactured by most of the larger producers.

With the growth of the automobile industry and the increasing popularity of the grease gun and high pressure lubricating equipment, the demand has arisen for a soft grease of great stability to withstand pressures of several hundred lbs. per sq. in.

Such greases are known as " pressure lubricants," and usually contain 10 to 15 per cent. of lime soap, a maximum of 1 per cent. of moisture, and an oil of viscosity that varies according to the manufacturer, from 300 to 500 secs. Saybolt at 100° F. Such greases are also excellent lubricants for ball and roller bearings operating under normal temperature conditions. They possess great stability and have little or no tendency to harden or change in characteristics in the presence of air and moisture.

For crank pin lubrication on slow-speed, heavy duty engines, and for certain steel rolling mill applications, it may be an advantage to use still heavier oils in the make-up of cup greases, and there are various products now on the market containing cylinder oils and heavy black oils of high viscosity.

Colouring and Perfuming Substances.

The use of colouring and perfuming substances in modern grease-making is rapidly decreasing. Previously their use was necessary to mask the objectionable characteristics associated with inferior

raw materials, but as it is now recognised that only the best qualities will stand up to present-day demands, the use of colours and perfumes has become unnecessary except for special requirements.

It is still customary to perfume greases to be used in food factories, where the odour of fats or mineral oils would be objectionable. In some cases slight colouring is resorted to in order to produce greases of uniform colour from raw stocks that are liable to slight variations in colour. Again, certain greases are coloured or scented to differentiate them from other products, and thus ensure that they may be easily identified. Soap dyes are those most commonly used where a colour is necessary.

Of perfuming substances used at the present time, nitrobenzene (oil of mirbane) is rapidly losing its hold in favour of perfumes of the essential oil type and synthetic so-called "deodorants."

Processes for the Manufacture of Cup Grease.

Cup greases may be manufactured in any of the following ways :—

1. In open steam-heated kettles.
2. In open kettles in which the source of heat is gas, oil, coke, coal or wood fire, or the circulation of hot oil or other heat carrying fluid, or by means of electrically-heated resistance coils.
3. By a two-stage process—*i.e.*, manufacture of a separate lime-soap to be used in subsequent grease-making operations.
4. In closed steam-heated kettles under pressure.
5. By the neutralisation of fatty acids with hydrated lime.

Manufacture of Cup Grease in Open Steam-heated Kettles. Manufacture of cup grease in open steam-heated kettles may be conducted, broadly speaking, in one of two ways :—

(*a*) The wet method.
(*b*) The dry method.

The Wet Method.—The fat, a small quantity of the mineral oil and the appropriate quantity of milk of lime are agitated in a steam-heated kettle until the saponification reaction is complete. The greater part of the excess moisture will have been driven off during this operation. In one variation of the process, heating and agitation is continued until the water content is known to be about right for the formation of a grease when the remaining mineral oil is added.

Others prefer to drive off all the moisture, cool the contents of the kettle by adding some of the balance of the oil, then adding the necessary amount of water to form a stable emulsion with the remaining oil.

The Dry Method.—The dry method of making cup grease differs from the wet method only in that dry hydrated lime instead of milk

of lime is put into the kettle at the start together with just sufficient water to enable saponification to proceed effectively. When saponification is complete the water and the mineral oil are added.

The dry method is quicker and more likely to give consistent results than the wet method. The time necessary to evaporate

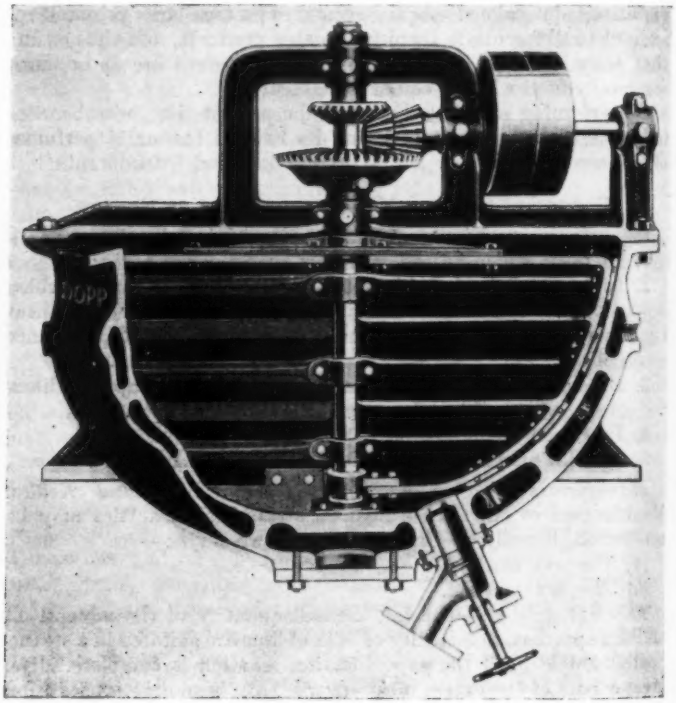

FIG. 1.

DOPP SEAMLESS JACKETED KETTLE WITH DOUBLE-MOTION POSITIVE-SCRAPING AGITATOR SUITABLE FOR BELT DRIVE.

large quantities of moisture is avoided. Furthermore, the exact poundage of lime necessary for saponification can be added to the kettle and the process accurately followed by withdrawing samples and estimating the excess lime. When this has vanished, or has reached a predetermined figure, it is time for the next operation.

Generally speaking for a 25–30 barrel batch of grease the wet method may require from 6 to 20 hr. per batch, while the dry method can usually be completed in 5 hr.

Manufacture of Cup Grease in Open Kettles other than Steam Heated.—The general procedure closely follows that outlined above, but it will be obvious that heating by means of direct firing renders

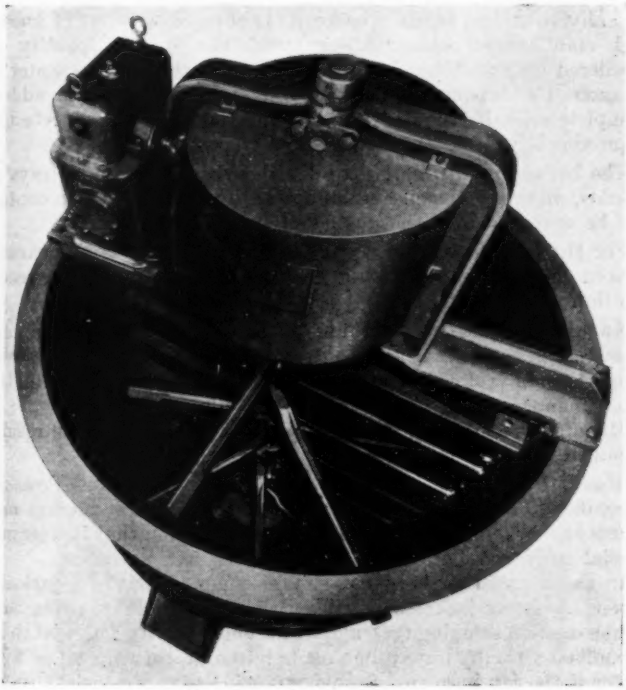

FIG. 2.

LOOKING DOWN INTO DOPP DOUBLE-MOTION SCRAPING TYPE MIXER WITH ELECTRIC MOTOR DRIVE.

temperature control in cup grease making more difficult, and great care has to be exercised to avoid burning any portion of the kettle contents.

Heating by the circulation of hot oil or other fluid is perfectly satisfactory and may even possess advantages over steam with regard to the rate of heat transfer and ease of manipulation.

Electrically-heated grease kettles are reported to exist, but have not to date been met by the author.

Manufacture of Cup Grease by a Two-Stage Process.—For manufacturers engaged in preparing large quantities of cup grease from one type of soap base this process possesses considerable advantages over those already described.

The first stage of the manufacture comprises the preparation of an anhydrous lime soap. The kettle is charged with fat of known and standardised characteristics and the exact quantity of powdered hydrated lime is added together with sufficient water to promote the saponification reaction. No mineral oil is added. Complete saponification of 1 ton of fat can usually be effected in approximately 1 hr.

The hot anhydrous lime soap can then be run out into trays or moulds, allowed to cool, and stored until required. Close control can be exercised over the quality of the soap produced.

For the manufacture of a cup grease, the lime soap is coarsely broken and preferably ground to a fine flour to aid its ready solution in the mineral oil.

An exact quantity of the lime soap flour is weighed out and dissolved in a convenient quantity of mineral oil. The correct proportion of water is added and the grease finished off in the usual manner.

Manufacture by this method is quick and exact, and the results obtained are very consistent.

Manufacture of Cup Grease in Pressure Kettles.—Pressure saponification is the most modern aid to grease production and differs so markedly from open kettle saponification that it warrants special consideration.

In the simplest, although not most common, use of a pressure mixer, a steam-jacketed kettle with pressure-tight cover and double-motion scraping type agitator is charged with : the fat to be saponified ; the hydrated lime made into a cream with oil or with water if the fat is so free from fatty acids that insufficient water will be formed by the reaction, and all of the lubricating oil needed to make the finished grease.

The pressure mixer is closed, the agitator started and steam turned into the jacket. After a predetermined time, the steam is turned off and the jacket vented and slowly filled with cold water; then, while agitation continues, cooling water is circulated through the jacket until the grease has cooled sufficiently to be drawn off without losing its moisture.

Advantages of this method lie in the fact that the whole process is standardized and mechanical, and is thus free from the un-

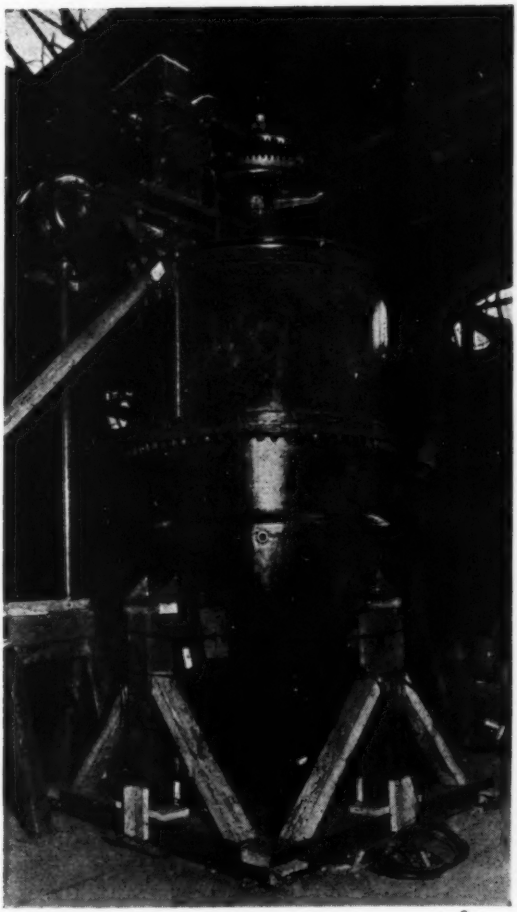

FIG. 3.

OPEN TOP DOPP GREASE KETTLE, CAPACITY 1420 IMPERIAL GALLONS, USED FOR MANUFACTURE OF AN EXCEPTIONALLY HEAVY GREASE WITH A DOUBLE-MOTION AGITATOR DRIVEN BY A 25-H.P. MOTOR.

certainty of the human element. By using the same raw materials, each batch of finished grease is exactly like each previous batch.

The disadvantages are that the quantity in each batch is limited to the capacity of the pressure mixer; that the presence of the

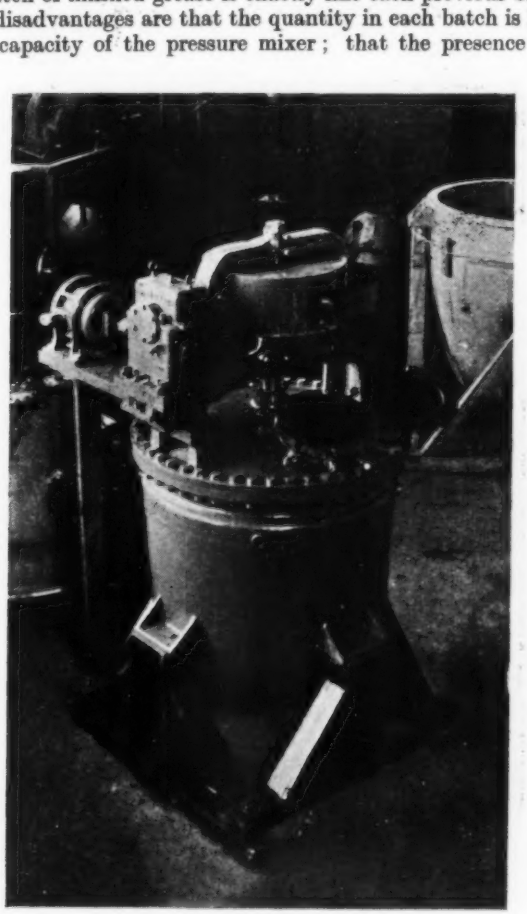

FIG. 4.

A 250 IMPERIAL GALLON DOPP PRESSURE MIXER WITH INDIVIDUAL MOTOR DRIVE.

large volume of oil needed for the finished grease slows up the saponification reaction and may necessitate the use of higher temperatures and pressures, and that certain oils are darkened

by prolonged heating. The procedure is also limited to the use of fats having a low acid value, for with fats that are high in free fatty acid an undesirable amount of moisture results from their neutralisation. With neutral fats, glycerine instead of water is formed, and so a definite amount of water can be charged in order to obtain a grease of a definite moisture content.

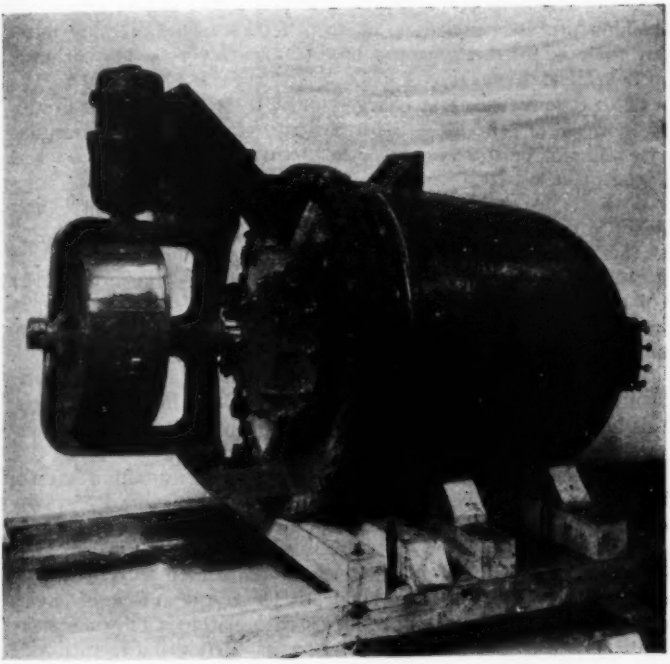

FIG. 5.

LATEST TYPE 250 IMPERIAL GALLON DOPP PRESSURE MIXER WITH INDIVIDUAL MOTOR DRIVE. THIS UNIT HAS A 5-H.P. HIGH-SPEED MOTOR AND SPEED REDUCER BUILT IN ONE UNIT AND MOUNTED ON A BASE CAST AS PART OF THE BRIDGE WHICH CARRIES THE LEVEL GEARS, ETC.

Actually, this method is normally confined to special products that present difficulties in their manufacture or standardisation.

A modification of the process just described is the use of the pressure kettle to prepare a lime base without oil being present. oil under pressure being added later and worked in before the

pressure is released. This works well with certain fats that are exceptionally difficult to saponify with lime in an open kettle or where saponification cannot be carried out properly when reacting agents are diluted by the oil, yet where the moisture content must be so carefully controlled that it is not practical to work the oil with the base in an open mixer.

The most generally accepted method of pressure-kettle working is to use the autoclave only for the preparation of the base, the finishing process being done in open kettles. One pressure mixer can then be used to supply base alternatively to two or more open kettles.

The pressure mixer is charged only with the fat, the lime, a little water and in most cases a little oil, so that the resulting base will not be too viscous or too hard to work into oil later on in the open kettles.

With steam at 125 to 150 lb. per sq. in. in the pressure mixer jacket, an internal pressure of 70 to 85 lb. per sq. in. is developed. At this pressure and temperature, saponification of the usual type of fat is 95 to 97 per cent. complete in 15 to 20 mins., and under 1 per cent. remains unsaponified at the end of 30 mins., but in production units it is customary to hold the batch at this pressure for 30 to 45 mins.

The base is then discharged into one of the open kettles, losing most of its moisture by "flash evaporation" in the discharge. This gives a nearly dry base to which definite amounts of oil and water are added for finishing.

Most installations have the pressure mixer mounted on the floor above the open mixers, so that the soap drains by gravity out of the discharge lines. A few installations have the top of the pressure mixer level with the tops of the open kettles, using the pressure generated in the pressure mixer to force the soap up into the tops of the open kettles. The disadvantage of having to remove soap from the lines after each batch is blown is more than offset by the convenience in operation resulting from having all the kettles on one floor.

In some cases the base is discharged directly below the surface of oil contained in the open kettle, and it is an advantage to have such oil warmed to a convenient working temperature, in order to avoid sudden chilling of the soap and the formation thereby of hard lumps.

Including the time required for charging and discharging the pressure mixer and for bringing the batch up to the correct pressure and temperature, a six-barrel batch of base is usually made in from 1½ to 2 hr. Finishing the grease in open kettles will probably require another 1 to 1½ hr., so that in practice the combination of

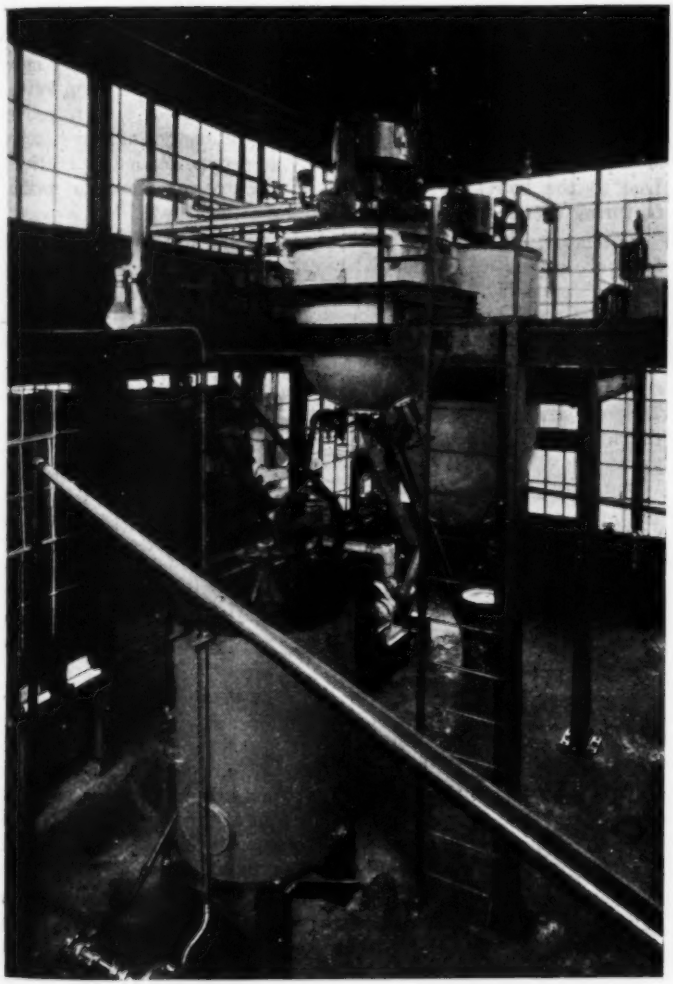

FIG. 6.

VIEW IN A GREASE PLANT SHOWING A 250 IMPERIAL GALLON PRESSURE MIXER ARRANGED FOR DISCHARGE INTO AN OPEN TOP MIXER OF 1420 IMPERIAL GALLON CAPACITY.

these operations results in two and in most cases three batches of grease from each open kettle per 8-hr. day, as compared with the older practice of making only one batch per kettle per day.

Grease from fats saponified under pressure is clearer and more transparent than when the saponification is done in an open kettle. Furthermore, the use of pressure saponification not only improves the uniformity of the grease but reduces the chance of lost batches and permits grease manufacture by less experienced operatives. Due to the increased throughput of the open kettles the manufacturing cost is also appreciably reduced.

The most popular sizes of pressure mixers are those of 300 and 500 gall. capacities, although they are made up to and including 800 gall. capacity. Probably the best known are the Dopp kettles made by the Sowers Manufacturing Company, of Buffalo. These kettles, built to withstand a test pressure of 300 lb. per sq. in., both in the jacket and in the interior, are of seamless jacketed construction which reduces the possibility of leaks to the minimum. They are fitted with cast-through type outlets which close flush with the inside of the kettle and so eliminate any pocket of unmixed grease.

Efficient and economical grease-making depends to a large extent upon the type of agitator used. The Dopp pressure mixers have two sets of paddles revolving in opposite directions at different speeds, and with adjustable spring steel scraper blades acting on the kettle wall opposite the sweep. The lower blade of the agitator just clears the bottom of the kettle and is slotted to clear a projection cast into the bottom of the kettle to permit the fitting of a thermocouple.

Manufacture of Cup Grease by the Neutralisation of Fatty Acids.—Although it is well known that both oleic acid and stearic acid can be used alone to manufacture cup greases, the use of stearines and the unpressed acid distillates of wool grease, cotton seed oil and fish oils, also the fatty acids from tallow, bone grease and even castor and linseed oils, would appear to be confined to this country. This is surprising in view of the simple type of equipment needed to produce remarkably good products therefrom.

Such materials are rarely used singly, but various combinations amongst themselves and even with neutral fats enable the user to produce certain specialised types of products that it is not possible to obtain by other means.

The process by which greases are produced from such starting materials is usually referred to as the " Cold Process " owing to the fact that direct neutralisation of the acidic material with hydrated lime is usually brought about at temperatures below the boiling

point of water. Boiling to produce saponification is unnecessary and in many instances detrimental to the finished product.

The usual method of procedure is to dissolve the selected fatty acid material or materials in approximately four-fifths of the total quantity of oil, and warm the solution to 120–150° F., and then to add a suspension of hydrated lime in the remaining oil, whilst gently agitating the mixture. Reaction in most cases is practically instantaneous and is accompanied by a rise in temperature.

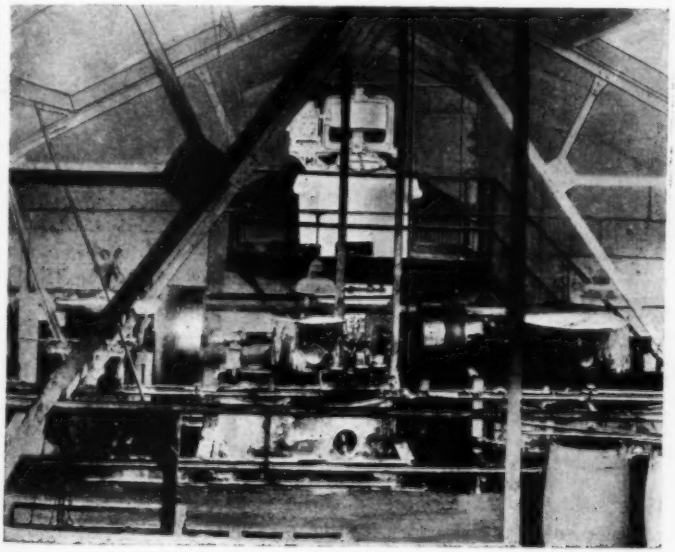

FIG. 7.

VIEW OF PRESSURE MIXER ARRANGED FOR THE MANUFACTURE OF BASE WHICH IS FINISHED IN OPEN TOP MIXERS SHOWN IN FOREGROUND.

Appropriate selection of the ingredients, and due attention given to the conditions of manufacture, especially with regard to the rate and extent of mixing will result in the production of grease of practically transparent appearance and remarkably low moisture content.

It is possible by this method to manufacture greases of a given consistency with less fat than would be required in the " boiled " grease process. Since a smaller quantity of lime is necessary to

produce the soap base the resultant greases will also be lower in ash content.

Greases produced by this method are remarkably stable under heat and pressure conditions. In some instances the soap appears to be in almost true solution in the mineral oil, since the grease can be heated as high as 250° F., and kept molten for quite appreciable periods of time, and yet on cooling will reset to almost the same consistency as that of the original product.

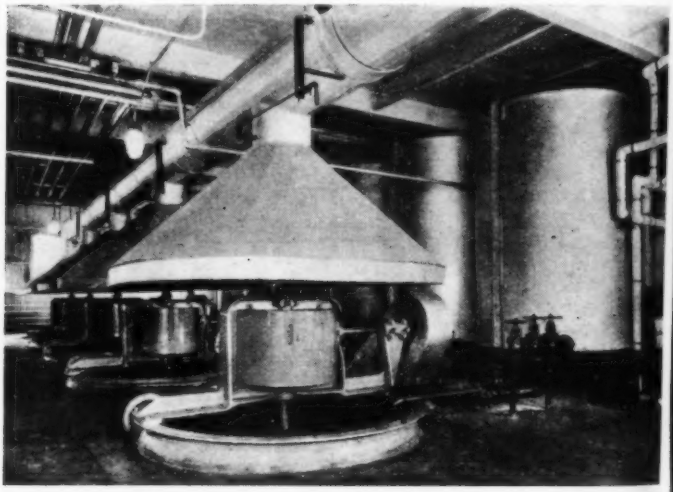

FIG. 8.

VIEW SHOWING TOPS OF DOPP OPEN TOP MIXERS IN A LARGE GREASE PLANT. THE AGITATORS ARE CONNECTED BY CHAIN DRIVES TO SPEED REDUCERS AND MOTORS SUPPLIED UNDER THE CEILING OF THE FLOOR BELOW. NOTE STORAGE TANKS FOR OILS, FATS, ETC.

Unfortunately, owing to the apparent simplicity of this method of manufacture, it is frequently misused by the less scrupulous manufacturer, with the result that his greases often contain excessive quantities of free lime or free acid, and suffer change of consistency during storage.

SETT GREASES.

The sett greases, which are mixtures of the calcium soaps of rosin acids and various grades of mineral lubricating oils, constitute the cheaper grades of semi-solid lubricants. They are important

in that they are satisfactory for the lubrication of rough machinery and heavy bearings operating at slow speeds. They are also extensively used for gear and wagon axle lubrication. These greases invariably contain an excess of the saponifying agent which, in the form of hydrated lime, is insoluble in the grease. This excess constitutes an abrasive element, and such greases are therefore unsuitable for lubricating accurately machined parts.

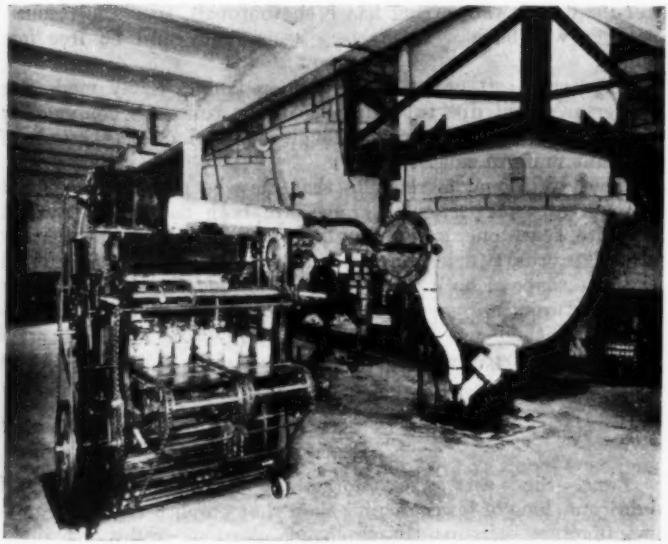

FIG. 9.

LOWER FLOOR OF GREASE PLANT SHOWN IN KETTLE IN FOREGROUND IS 1420 IMPERIAL GALLON CAPACITY, THE OTHERS ARE OF 833 IMPERIAL GALLON CAPACITY. TWO OF THE KETTLES ARE DIRECT FIRE-HEATED, THE OTHERS USE STEAM AT UP TO 150 POUNDS PER SQUARE INCH GAUGE PRESSURE. THE GREASE IS DRAWN FROM THE OUTLET AT THE BOTTOM OF A KETTLE, PUMPED THROUGH A SCREEN AND THEN TO AN AUTOMATIC TIN FILLING MACHINE.

Sett greases are almost invariably made by mixing while cold, or at a temperature below 150° F., mineral lubricating oil, rosin oil and a lime mixture known as "sett." The sett is normally an emulsion consisting of mineral oil, water, hydrated lime, and a small quantity of the calcium soap of rosin acids which promotes emulsification. In certain circumstances, the sett may simply

comprise a suspension of finely divided hydrated lime in a mineral oil of low viscosity. In both cases, if the lime is not held in a fine state of division in the emulsion, the reaction with the major portion of the rosin oil in the final mix will be uneven and slow, resulting in a lumpy grease and one which is prone to separate.

The final mixture will normally solidify in a few minutes, and in some cases in less than a minute, and providing that proper precautions have been exercised in the selection of the various stocks, that the ingredients have been mixed in the correct proportions and that the mixing has been thorough, then the resultant product will have a satisfactory consistency and be free from lumps.

In general, sett greases are considered more easily made than cup greases, but due to variations in the rosin oils commercially obtainable, it is sometimes difficult to produce sett greases of constant uniform consistency.

Other additional ingredients that may be present in cold sett rosin greases of particular grades are both flake and powdered graphite, soapstone, gypsum and similar fillers, whilst occasionally other fats or fatty acids may be added, in which case there is formed the lime soaps of both the fatty material and the rosin oil. Some manufacturers use small quantities of caustic soda in the manufacture of their sett greases, resulting in products which are of particular value where a grease is required to adhere well to wet metallic surfaces.

Raw Materials Used in the Manufacture of Cold Sett Greases.

Lime.—Since sett greases normally constitute a rougher class of lubricant, lime of lower quality than that purchased for cup grease manufacture is frequently employed. Various grades of hydrated lime are available, and a considerable quantity of rock lime, which has of course to be slaked before use, is still employed. The slaking may be done by hand, but it is preferable to use mechanical apparatus that slakes the lime, removes by centrifugal action the grit and impurities always associated with rock lime, and delivers through a fine gauze screen a milk of lime suspension of fairly regular composition.

Rosin Oil.—Rosin oils can be purchased in many grades, each differing from the other in viscosity, colour and free acid content. Rosin oils used in the manufacture of sett greases are a source of considerable variation in the consistencies of the finished greases, and it is possible for the usual laboratory tests of an oil to be practically identical with those of a previous supply found satisfactory and yet for the oil to be inferior in its grease-making qualities. Since at

the present time it seems impossible adequately to specify in terms of laboratory tests the desirable qualities of a satisfactory oil, it is customary for the rosin oil manufacturer to submit a sample of the product he intends to supply and for the grease maker to determine its grease-setting properties by making small experimental batches, using the ratio of mineral oil and sett to rosin oil that is used in the grease plant. Any specification for rosin oil should include limits for :—

Gravity.
Colour.
Viscosity.
Acidity.

In addition, the oil must be a pure rosin oil, unadulterated and free from impurities.

Colour is important where light coloured greases are being manufactured.

Rosin oils with satisfactory grease-making qualities usually have a Redwood viscosity at 200° F. exceeding 200 secs.

Rosin oils of high abietic acid (molecular weight 346) content are usually though not invariably of greater value in regard to their grease-setting properties. A good quality oil averages about 50 per cent. acidity.

Light axle greases are made from a pale, low viscosity mineral lubricating oil, and are practically anhydrous, whereas tram or tub greases are made from black oils and usually contain both water and fillers.

Sett greases have higher melting-points than the cup greases and, like them, are insoluble in water. When manufactured from good-quality mineral oils and light coloured rosin oil, they are pale in colour and closely resemble the cup greases in appearance, for which for the rougher type of lubrication they may form cheaper alternatives.

By far the larger quantity of sett greases are made from black and waste oils, and contain some cheap filler and up to 30 or 35 per cent. of water.

Floating grease, corfe grease, curve grease, tram grease, axle grease, wheel grease, wagon grease, tub grease and skid grease are all names given to varieties of sett greases.

2. Soda Base Greases.

These products are mixtures of mineral lubricating oil and the sodium soaps formed by the saponification of any of the natural fats or fatty oils with caustic soda.

In those greases containing water, an emulsion of the oil in water type exists. However, many soda soap greases are more or less

completely dehydrated and are not emulsions but intimate mixtures of soap and oil. From a practical consideration, they are mineral oils sorbed by a lattice structure soap, giving a plastic solid.

Soda soap greases may readily be recognised by rubbing a small portion in the palm of the hand with water. The appearance of the grease is changed and the water soluble soap that is present is readily seen. In other words, the soda soap greases disintegrate in the presence of water.

Much of what has been said in regard to lime soap greases applies also to soda soap greases. The same types of saponifiable oils and fats may be used. Similar methods of manufacture and similar types of equipment may be employed. Also, the remarks made concerning the quality of the mineral lubricating oil used apply equally to soda soap greases. However, soda soap greases differ markedly from the lime soap greases with regard to the number of physical forms in which they can exist. Whereas lime soap greases, by whatever method they are manufactured, are characteristically buttery, short textured products ; this is not so with the soda soap greases. Changing the method of manufacture will produce entirely different types of greases from the same raw materials.

Soda soap greases have a higher liquefaction point than any other soap-oil mixture, and show less change of consistency over the temperature range approaching the liquefaction point.

Due to their solubility in water, however, their use is limited to conditions where moisture is absent or where it is definitely desirable to form a lubricating cream by the action of moisture on the grease.

The Manufacture of Soda Soap Greases : Cold Process Method.— Smooth, short texture, buttery greases, similar in appearance to lime soap cup grease, can be manufactured by neutralising fatty acids dissolved in a low viscosity mineral oil with a strong aqueous solution of caustic soda. Very efficient agitation is essential to ensure that hard lumps of soda soap do not form, and the reaction temperature should be kept as low as possible.

By a similar method of manufacture with specially chosen fatty acids and working at a slightly higher temperature, practically transparent smooth buttery greases of the " solidified " oil type can be produced.

Making Soda Soap Grease in Steam Jacketed Open Top Kettles.— For this type of operation only the lower portion of the kettle needs to be steam jacketed. Above the jacketed surface a large non-jacketed volume is desirable to provide room for foaming during saponification and while the moisture is being driven off. In consequence, it is often economical to provide a full jacketed kettle

with an upper welded belt to provide the foaming or grease thinning space.

In operating this process, it is usual for the kettle to be charged with the saponifiable fat and an approximately equal quantity of the mineral oil. After melting the fat and thoroughly mixing, the full quantity of caustic soda for complete saponification is added in the form of a solution in water, the strength being adjusted to the type of fat employed.

Steam is turned on in the jacket of the kettle and the batch is cooked while stirring. A steam pressure in the jacket of 125 to 140 lb. per sq. in. is customary. As a general rule, further additions of oil to the mixture is made slowly as soon as the soap commences to wind itself round the agitator.

Making Soda Soap Grease in Direct-Fired Open Top Kettles.—In the early days of the grease industry it was customary for all boiled soda soap greases to be fire cooked, and many practical men still prefer this method. The custom probably arose because it was not practicable to use the steam pressures of 100 to 150 lb. per sq. in. that are common to-day. As a result soda soap grease then made in steam jacketed kettles contained undue amounts of moisture which could not be removed at the low temperatures obtainable. Thus the better soda soap greases then made were prepared in fire heated kettles in order to obtain the temperatures necessary for practically complete dehydration.

At the present time, with efficient agitators, jacketed kettles have the advantage that uniform temperatures throughout their contents are more readily obtained than with fire heated kettles, where hot spots quite often cause trouble. For really high temperature requirements, hot oil circulating through the jacket will provide any temperature needed.

On the other hand, where it is necessary not only completely to dehydrate the grease but to drive it to a liquid, modern practice still employs the direct heated kettles usually gas-fired.

It is desirable for the kettle wall to be a smooth, true surface that permits the use of a scraping agitator. Such scraping materially shortens the time required for the contents to reach a given temperature and helps to prevent local overheating and burning of material that might stick to the kettle wall without such scraping.

This type of kettle is particularly suitable for those products that need to be heated to a final temperature of 450–500° F.

Making Soda Soap Grease in a Steam Jacketed Pressure Mixer.—The manufacture of a batch of soda soap grease in an open top steam jacketed kettle is usually a full day's operation and may

even take longer. The use of a pressure mixer will at least halve this time.

For a batch of soap it is customary for the pressure mixer to be charged with the saponifiable fat, the caustic soda as a 33–50 per cent. solution, and a small quantity of mineral oil. Two hours is usually sufficient for the charging of the mixer, the saponification reaction and the discharge of the base.

The pressure mixer retains in the soap the moisture from the caustic soda solution as well as any liberated from the reaction. Part of this moisture is dissipated by "flash" evaporation when the soap base is being discharged from the pressure mixer into the open kettle. The remaining moisture is removed in the open top steam jacketed kettle where the soap base is agitated for one hour.

The remaining oil is then slowly added and worked into the batch in the same manner as when the grease is made entirely in the open kettles.

After the pressure mixer has been discharged, live steam is blown through it and through the distributing pipe to remove any soap which might otherwise accumulate. The pressure kettle requires no further cleaning.

Types of Soda Greases.—Mention has already been made that a typical cup grease can be made by neutralising in the cold a solution of fatty acids in a mineral oil with a caustic soda solution.

By the application of slight heat to such a grease a perfectly transparent product still of the same buttery consistency can be prepared.

Still stronger heating will transform the grease into a much stiffer spongy or fibrous type of material of definitely higher melting-point and more or less completely dehydrated.

Sponge or Fibre Greases.—The term sponge grease or fibre grease is usually applied to soda base greases of medium consistency. The fibre texture of the grease depends upon the degree of dehydration of the soap, the proportion of soap and the nature and viscosity of the mineral oil, the quantity of glycerine remaining in the grease and the method of mixing. It is not due, as is commonly supposed, to the addition of fibrous filling matter.

The sponge greases have high melting points and are used primarily where bearings run at elevated temperatures due to radiated or conducted heat. The softer and medium consistency greases are suitable for use through screw-down grease cups.

They do not separate readily from the mineral oil, but give stable and thick, tenacious lubricating films.

These greases are usually manufactured from tallow or similar type fats and lubricating oils of from 200 to 1500 secs. Redwood

viscosity at 70° F. in an open steam jacketed type kettle or combination of pressure mixer and open type kettle.

Gear Greases.—Soda soap gear and transmission greases are really sponge or fibre greases of relatively low soap content made with a high viscosity oil. The heavy mineral oil and the fibrous texture of the soap make the product very tenacious.

Gear greases vary in consistency from fluid to semi-solid. The gear speed is of prime importance in determining the grade of lubricant to be used. Whether the speed be high or low, the grease must possess pronounced cohesive property to enable it at all times to follow in a stream with the moving gear and a readiness to flow that will prevent channelling. It must be sufficiently fluid to give the minimum resistance to the turning of the gears, and it must be perfectly stable and not change its consistency or separate when exposed to agitation or heat.

Gear greases are manufactured in the same type of equipment as is used for sponge greases. The use of a high viscosity black oil and rapid cooling after saponification are necessary in order to produce a grease of smooth texture.

Soda Soap Solidified Oils.—Solidified oils are usually made in single walled fire-heated kettles. The fats used are selected to produce a soap stock more than usually soluble in mineral oil, and one that does not produce a coarse, fibrous texture when the solution is cooled to normal temperatures.

The fats selected, the choice of mineral oil, the ratio of caustic soda to fatty ingredients, the rate of heating and the rate of cooling are all very important factors in the production of these smooth unctuous greases.

Saponification is carried on in much the same way as in making other soda greases, with the exception that after all the oil is in, the temperature of the batch is rapidly raised to 400–450° F. The fire is then turned out and the temperature allowed to drop to about 350° F. The grease is then run into shallow pans to a depth of 4–5 in.

The stiffer types of grease after setting are cut into candles, squares or blocks, and wrapped in oil-proof paper. If a smooth buttery grease is required, the cooled grease may be run through a mill.

Soda soap tunnel bearing grease is a very hard lubricant manufactured as above, and intended for the lubrication of the tunnel shaft bearings of ships. It is not melted at the usual operating temperatures and gives a good lubricating film in the presence of sea water.

Hard soda soap grease blocks are frequently used on the open type bearings of the dryer rolls of paper machines, beaters and

calenders, and are frequently prepared from high-grade filtered cylinder oils and a previously prepared soda soap. These greases are completely dehydrated, and finally heated to a temperature of 450 to 500° F. in a gas fired kettle.

The soap stock and mineral oil must be so chosen and proportioned that in use the grease block maintains in contact with the bearing a soft oily surface that possesses no tendency to harden or carbonise. It must also wear away sufficiently rapidly to keep itself free from accumulation of fluff and dirt which are always present in the surroundings and which, if allowed to collect, would help to impart to the grease block a hard glazed surface.

Railway Greases.—Lubrication by greases is to all intents and purposes non-existent in British locomotive practice, but in North and South America, in India, and to a certain extent in South Africa, China and Australia, grease lubricated locomotives have superseded other types.

Grease lubrication is not, of course, applicable to the valves and cylinders of a locomotive, nor yet to the axle-boxes, other than those of the coupled wheels, but crank pin bearings operating under high temperatures, and axle-box bearings the journals of which run at a high peripheral speed can be grease lubricated.

For driving journals an exceptionally hard soda soap grease having an abnormally high melting point and a plastic consistency is made.

Although exceptionally hard, the grease must not be dry or crumbly, and must be capable of being moulded under pressure into cakes which are fitted into a cellar and held by means of a loaded spring hard up against the journal face.

If an axle-box lubricator is properly fitted and suitable grease applied, the bearing should run 10,000 to 20,000 miles without attention other than occasional inspection.

A slightly softer grease of the same type is used for crank pin bearings. The close fit of these bearings and the hardness of the grease have led to the development of a special grease gun that is used in conjunction with nipples screwed into the cavities of the connecting and coupling rods, and with which a pressure of 5000 lb. per sq. in. can be exerted, thus ensuring the penetration of the grease to every part. For this service the grease is moulded into candles of a size corresponding to the bore of the grease gun.

The mileage between greasing on a locomotive equipped for grease lubrication averages 1000, and although it is doubtless true that a certain frictional loss is entailed through the use of grease, this is more than offset by the definite decrease in labour and upkeep costs.

The principal requirement of this class of grease is that it must maintain its consistency and body at the operating temperatures, usually about 250° F., and yet feed on to the bearing properly and possess no tendency to carbonise.

Earlier greases for this purpose were tallow compounds and semi-fibre greases made by the action of caustic soda on fats or fatty acids in the cold or substantially under the boiling point of water.

Due to the fact that they were not boiled, the fibre formation was not pronounced and they had a texture midway between cup greases and the usual fibre grease.

Owing to their method of manufacture, however, such greases created trouble by their moisture content causing frothing and foaming at operating temperatures. Also, the excess of caustic soda necessary to drive the saponification to completion caused burning to the hands of the operatives.

The best types of locomotive driving journal compounds and side rod cup greases now available are heavily boiled and completely dehydrated soda soap greases free from any deleterious caustic soda. They have remarkably high melting points and retain their hardness and consistency practically unchanged up to temperatures of 200 to 250° F. The actual melting point of such greases is usually above 400° F.

Railway Wagon Axle Greases.—In this country oil lubrication is almost universal for main line rolling stock, but many goods wagons, colliery wagons, etc., are still grease lubricated.

Where such wagons run over main lines the railway companies insist on the use of a lubricating grease to the Railway Clearing House Specification, which states that "the grease is to be an emulsified mixture of water, soda soap and neutral tallow or other suitable fat, with or without mineral oil. It must be free from resin or tar oil, and also from lime, gypsum or other mineral matter, and must not contain more than 50 per cent. of water." A certain heat test is prescribed, and the specification concludes by stating that greases of the following composition have proved satisfactory in use :—

Tallow or other suitable fat	15–25 per cent.
Mineral oil (not more than)	20 ,,
Dry soda soap	15–25 ,,
Water (not more than)	50 ,,

In one method of manufacturing a grease to meet the Railway Clearing House Specification, a palm oil-soda soap is allowed to soak in water until thoroughly softened. The mixture is then brought to a temperature of about 150° F., the moisture adjusted

to give the required proportion in the finished grease and the appropriate quantity of tallow added and thoroughly stirred in. Finally a heavy petroleum residuum is added, and the mixture is then run off into pans and kept agitated until set.

Unfortunately a good many greases that meet the Railway Clearing House Specification are very inferior in quality, and are fit only for the roughest type of colliery or quarry truck lubrication.

3. Lead Soap Greases.

Lubricants containing lead soaps have been used to a small extent for many years for various purposes, as, for instance, railway car journals, but the trend of recent developments in the automobile industry has brought about increased popularity and study of these products.

In well lubricated bearings the loads rarely exceed 2000 lb. per sq. in., and the rubbing speeds are generally high enough to maintain a film of oil which separates the bearing and the journal. However, in the modern automobile gears the pressures between gear teeth may reach values as high as 40,000 lb. per sq. in., with rubbing velocities ranging from 0 to 300 ft. per min. and operating temperatures of 210° F. With such conditions prevailing it is not difficult to see that the limit of the load carrying capacity of ordinary oils and greases will have been passed.

Investigations have been carried out in particular by the General Motors Corporation, the Gleason Gear Company and the Timken Roller Bearing Company, with specially designed apparatus for measuring the load carrying capacity and frictional properties of both oils and greases, and it has been shown that the addition of certain types of lead soaps to lubricating oils considerably increases their film strength.

Lead soap added to sulphur-free mineral oil does not seem to increase its load carrying ability appreciably, but lead soap added to a mineral oil containing sufficient sulphur will produce an "extreme-pressure" lubricant that will maintain an effective film with less friction and heat than a sulphurized mineral oil.

In addition to their pressure-resisting properties, lead soap lubricants possess other characteristics peculiarly their own.

The addition of a small quantity of a lead soap to an oil will usually produce a considerably reduced cold test. Furthermore, a grease or oil of a given consistency will absorb a larger percentage of lead soap as compared with lime, soda or aluminium soaps. For instance, a 200 sec. viscosity mineral oil, when combined with 15 per cent. of lime soap, results in a cup grease of medium consistency. With the same oil 15 per cent. of soda soap would give a medium sponge grease, and 15 per cent. of aluminium

stearate a tough transparent grease of hard consistency. On the other hand, the addition of 15 per cent. of lead soap to the same grade of oil would result in a perfectly free-flowing lubricant.

This property of lead soap enables the manufacturer to reduce the amount of mineral oil in a given consistency, and to employ a larger amount of that part of the lubricant most effective in resisting pressure. Many lead base gear greases on the market contain, in addition, lime, soda or aluminium soaps solely for the purpose of obtaining a given consistency.

Lead soap lubricants do not emulsify nor dissolve in water. On the other hand, moisture has the effect of causing slight hydrolysis of lead soaps when in dilute solution in mineral oils, and lubricants containing them should be kept under moisture-free conditions.

Manufacture of Lead Soap Greases.—Two methods of manufacture are employed for lead soap greases—namely, direct saponification and double decomposition.

In the direct saponification method, the fat or fats, usually oleine or a mixture of fish oil or other liquid fat and oleine, is heated with litharge in an open gas-fired kettle, the temperature of saponification being in the neighbourhood of 475 to 525° F. When saponification is complete, the appropriate quantity of mineral oil is added.

The objection to this process is that invariably an amount of uncombined lead oxide remains in suspension in the finished grease to precipitate slowly therefrom afterwards.

The manufacture of lead soap lubricants from previously prepared lead soaps is preferred by most makers, as the resulting products are entirely free from suspended solids.

This method simply comprises the dissolving of a certain amount of the lead soap in a selected mineral oil.

Lead soaps, particularly lead oleate and lead stearate, are available in a satisfactory state of purity, or they can be prepared by the double decomposition method from a large number of readily available saponifiable oils and fats.

The more commonly used raw materials are whale oils, fish oils, oleines and stearines, linseed oil, castor oil, neatsfoot oil, nut oil, rosin oil, rosin and naphthenic acids. From any one, or a mixture of two or more, of these fats an alkali (usually the soda) soap is prepared. Having obtained an entirely satisfactory soap in aqueous solution, a solution of a water-soluble lead salt, usually lead acetate, is slowly and carefully added to the alkaline soap solution. Double decomposition occurs and the water-insoluble lead soap of the original fat or mixed fats is precipitated. It is purified by washing with hot water, after which it is freed from

excess water, and can then be used at once or completely dried and retained for future use.

Lead soaps are not entirely soluble in all mineral oils. Some lead soaps are more soluble than others, and only a limited number of mineral oils are suitable for use in lead soap lubricants.

Much of the trouble experienced with lead base lubricants, either from separation or from undue wear of the bearing surfaces, is caused by the use of unsuitable oils in their manufacture. Cheap black oils of low viscosity are quite unsuitable.

In the manufacture of lead base lubricants the greatest possible care must be paid to the purity of the lead soap, the type of fat or fats chosen, and the suitability of the mineral oil not only with regard to the use to which it is to be put, but also to its property for holding the lead soap in solution.

4. Aluminium Base Greases.

As ingredients in grease manufacture, aluminium soaps are gaining in popularity now that intensive study has revealed the best means for their utilisation.

Hitherto they have presented a most perplexing problem to the grease maker owing to the fact that every ingredient apparently plays a definite part in shaping the physical properties of the resultant product.

The aluminium soap of stearic acid, when incorporated with a low viscosity mineral oil, yields a grease of low cohesive character, as evidenced by its short shear and buttery texture.

The aluminium soap of oleic acid, incorporated with a similar oil, yields a product of high cohesive character and pronounced rubbery texture.

When incorporated with a high viscosity oil, aluminium soap yields a grease of softer consistency than when incorporated with a low viscosity mineral oil.

The rate of cooling has an important bearing on the grease texture and consistency, as also have the method of dissolving the soap, the rate of heating and the maximum temperature to which the oil and soap are heated. Important, also, is the actual chemical composition of the soap, the presence or absence of free fat and the character of the mineral oil used.

It is necessary for the grease manufacturer to study all these factors in order to arrive at the conditions best suited to the grade it is desired to market.

When properly made, aluminium soap greases possess all the favourable characteristics of lime soap greases. They are usually brilliant and transparent in appearance, and are often referred

to as solidified oils or transparent cup and pressure gun greases. They are stable at high temperatures.

Aluminium soaps containing a small amount of mineral oil, which is used to coagulate them during manufacture, are termed "pulp stock."

Mineral oils thickened by the addition of small quantities of pulp stock are often called mineral castor oils.

Aluminium soap greases are invariably prepared by dissolving the previously made soap in the selected mineral oil.

The aluminium soap is usually made by the double-decomposition method, in which a solution of a water-soluble alkaline soap is treated with a solution of aluminium sulphate or alum. The precipitated soap must be well washed to free it from impurities and is then dried, or may be immediately dissolved in oil.

The aluminium soap may also be prepared by the action of gelatinous aluminium hydroxide on the fatty acid.

One of the main objects in the renewed study of aluminium soap products has been the desire for an all-purpose automotive grease. This has resulted in the marketing of aluminium base, high-pressure lubricants, sometimes called "lucid compounds."

These consist, for the most part, of an aluminium soap dissolved in a medium motor oil, and are characterised by extreme stringiness and adhesiveness.

In colour they resemble transparent grease, having the bloom and opacity of the lubricating oil used in their manufacture.

Their stringiness is evidenced by their tendency to stretch out in long threads. They adhere tenaciously to any surface with which they come in contact and their consistency is such that they flow like oils.

Aluminium soaps possess the disadvantage of being hydrolysed by water, and this factor limits the application of aluminium soap greases to moisture-free conditions.

5. Special Types of Greases.

Mixed Base Greases.—The lubricating greases so far dealt with have all been straightforward single base greases, but it is no uncommon thing to meet greases which contain combinations of soap made from different saponifying bases. By such means certain specific features of one type become superimposed upon or associated with those of quite a different type.

At one time it was common practice in the manufacture of lime soap cup greases to use slightly less than the theoretical quantity of lime for complete saponification and to finish off the reaction with a trace of caustic soda.

Undoubtedly it was noticed that the addition of caustic soda tended to increase the melting point of the resulting cup grease and also to make the cup grease less water repellent and thus more adaptable for use on bearings operating in moist surroundings.

Appreciable quantities of soda grease can be incorporated in a lime soap grease without altering the smooth, buttery texture usually associated therewith. At the same time, the melting point is raised and the grease becomes characterised by the stability and non-separating properties of a soda soap.

The reverse is also applicable. Soda soap greases tend to be drier and less oily than lime greases, but the addition of a lime soap grease to a soda soap grease will counteract this property. Also with sponge greases, an addition of a lime grease will have the tendency to shorten the fibres, which may be an advantage under certain conditions.

Lime-soda and soda-lime block greases are by no means uncommon.

Potash soap greases are rarely met with as they tend to be altogether too soluble in water, but where it is desirable to obtain a high melting point grease of a relatively soft consistency, potash may be used in conjunction with caustic soda as the saponifying agent.

Calcium-potash soaps have also been met.

Lead and aluminium soaps are quite frequently associated with either lime or soda soaps to impart, in the first instance, greater pressure-resisting properties, and, in the second, greater stringiness, adhesiveness or clarity.

Mixed base greases may be made by any of the following methods : (*a*) by first preparing each soap in a separate kettle and then mixing the soaps and oil in a further process ; (*b*) by manufacturing both soaps simultaneously ; (*c*) by double decomposition preparation of a mixed-base soap, subsequently dissolved in oil.

Zinc Base and Zinc Oxide Containing Greases.—Zinc soaps are rarely incorporated with mineral oils to produce greases without the addition of other soaps, as they have little ability to thicken the oil. Their chief purpose is to modify the properties of soda soaps to give a smoother grease with better lubricating properties.

However, mention must be made of an interesting range of proprietary lubricants that contain considerable proportions of zinc oxide. These products consist of special types of oils and greases in which zinc oxide has been incorporated by a special process.

They were evolved to overcome a corrosion problem occurring under conditions of rapidly varying local stresses, and in this

connection were apparently successful. In practice it was found that with such lubricants the working parts became plated with a thin film, claimed to be metallic zinc, of an estimated thickness of 0·0004 mm.

This film, it is considered, is constantly being deposited and oxidised off again, with the result that the oxidation is confined to the zinc film and does not extend to the steel parts below.

Barium Base Grease.—A recent patent (E.P. 398,402) claims the use of a soap in the preparation of a consistent grease made by saponifying a fatty substance with crystallised barium hydrate. Colza oil, tallow or a mixture thereof is heated in an autoclave under pressure with the calculated amount of crystallised barium hydrate and the resulting soap is dissolved or emulsified in mineral oil to form a grease.

Miscellaneous.

Many instances occur where additional materials are added to the more usual type of greases to meet certain specific requirements.

Graphite Grease.—Graphite, in flaked, powdered or colloidal form is by no means an uncommon ingredient in lubricating grease. It can be incorporated in practically any type of grease and such lubricants find a wide variety of uses. Graphited greases are of especial value where the lubricant is liable to be washed away by water, oil or chemical solvents. Hydraulic rams, plungers, slides and bearings exposed to water, heavy driving chains for sprocket gears, and motor car springs, are typical instances where these lubricants are normally employed.

Hair and Wool Greases.—Animal hair, wool strands and cotton fibres are incorporated in certain types of greases for use particularly on heavy duty bearings in industries where dusty surroundings and general adverse conditions prevail, for instance, on ball and tube mills and on the carrying rollers of rotary kilns at cement works. Cleaner and more economical running is obtained by this method, as the fibrous grease prevents, as far as it is possible, the ingress of dirt and grit.

Such greases also find application for line shafting bearings in textile mills and foodstuff factories, and for certain types of high-temperature bearings.

The manner in which this type of grease is packed into a bearing is of greatest importance. Care must be taken to work any loose ends of the fibrous material into the grease, as if a strand were drawn in between the journal and its bearing there would be danger of the packing being displaced.

Waterproof Grease.—A product especially suitable for use on bearings subject to immersion in or prolonged contact with water, as on dredges, may be produced by the addition of a highly asphaltic residue or bitumen to a lime soap cup grease.

Liquid Greases or Non-Fluid Oils.—These are mineral oils thickened with small quantities of soap, usually lime, soda or aluminium. They possess the advantage that the lubricant has little tendency to creep over the shaft or to be thrown on to floors, adjacent woodwork and material in the process of manufacture.

Characteristics of Greases and Methods of Testing.

The principal characteristics of grease which are of interest to the manufacturer and user are as follows :—

1. Consistency and hardness.
2. Melting point (or flow point).
3. Stability : (*a*) in storage ; (*b*) in use.
4. Colour and odour.
5. Load-carrying capacity and other specialised properties.

1. Consistency and Hardness.

In selecting a grease for a definite type of lubrication, it is essential that the consistency be carefully considered. Where the grease has to be fed through long lengths of piping, a grease of a short texture should be chosen, as a grease of fibrous or rubbery consistency would offer too much "drag" or resistance to flow. An open bearing may be well adapted for lubrication with a dehydrated grease block, and in all cases effective lubrication and minimum consumption will depend to a great extent upon the consistency of a grease.

With oils consistency would be defined by viscosity, and with greases at the present time the term consistency may be regarded as "the degree of solidity" of a grease (Klemgard), and embraces both texture and hardness.

Various methods have been proposed and many instruments devised for determining the consistency of particular types of grease or for determining consistency in terms of some particular property of the lubricant.

Porter and Gruse[1] have described the determination of the consistency of cup greases by means of a plastometer, in which the time rate of flow through an orifice of definite size may be measured at different pressures.

When dealing with oils, however small the pressure, some flow will take place at a rate proportioned to the pressure, but with

[1] *Ind. Eng. Chem.*, 1925, **17**, 853.

greases, at small pressures, no flow may take place at all. If, however, grease is subjected to a gradually increasing pressure, a point will be reached at which it begins to flow, and from then on the flow rate will rapidly increase.

To determine the relation between the pressure and the flow rate for a grease, tests must be made at more than one pressure, since a determination made at one pressure will not permit predicting the flow rates at other pressures.

Knopf[2] describes means for evaluating various types of consistency.

For semi-fluid greases, such as gear and universal joint lubricants, it was found that the Saybolt-Furol instrument could be used with accuracy, but for plastic greases the Sinclair pressure viscometer was employed to measure the consistency in terms of viscosity.

The Sinclair viscidometer is an instrument developed primarily to determine the viscidity of plastic or semi-fluid lubricants by measuring the resistance offered by the passage of an object through the body of the lubricant, this being indicative of the drag resistance as well as the non-channelling characteristics of the product. The device consists of a constant velocity of descent of a spike-shaped plunger through a well-insulated jar of accurate bore containing the material to be tested. A weighing device reads viscidity direct at definite positions above and below the mid-travel position of the spike head.

The viscidity is defined as the average resistance offered by the lubricant to the passage of the spike through it. The test is of particular value as a control in the manufacture of gear lubricants.

A torsion viscometer of the MacMichael type can be used for measuring consistency or firmness, which is a function of the internal cohesiveness of a plastic lubricant.

Merrill[3] describes the use of the Abraham consistometer for measuring the consistency of cup greases. In this instrument the force necessary to cause a plunger to travel through the grease at a standard rate is measured. The results are reported as cube roots of the pressures and are known as Abraham hardness numbers or Abraham consistencies.

A new type of pressure viscometer in which the test material may be passed successively back and forth through a capillary, thereby giving consistency readings which will show whether the material is changing under stresses, is described in Research Paper No. 188 of the American Bureau of Standards, July, 1930.

[2] *Ind. Eng. Chem., Anal.*, 1934, **6**, 126.
[3] *Ind. Eng. Chem.*, 1925, **17**, 1068.

Undoubtedly the most common method of determining the consistency of cup greases is by the use of some form of penetrometer.

By the Air Ministry method, a steel tube $\frac{13}{16}$ in. in diameter and $2\frac{1}{2}$ in. long is filled with grease and placed in a graduated glass tube 9 in. long, the lower end of which is closed with a tightly-fitting cork. After being maintained at a temperature of 18° C. for 30 minutes a graduated glass guide tube 33 in. in length and of about $\frac{7}{16}$ in. internal diameter, held vertically in a retort stand, is inserted concentrically in the first glass tube so that its lower end is 1 in. above the surface of the grease.

A cylindrical aluminium rod $\frac{3}{8}$ in. in diameter, of known length, weighing 18 grams, flat at its lower end and supported by a thread through a hole in its other end, is allowed to fall down the guide tube from a height of 10 in. above the grease surface. The depth to which the plunger penetrates is measured immediately after the drop.

Probably the most widely adopted method for testing consistency is the penetration test proposed by the American Society of Testing Materials (Report of Committee D-2).

The apparatus employed is a standard asphalt penetrometer in which the needle used for asphalt penetration tests is replaced by a stainless steel or brass cone with a detachable hardened steel or stainless steel tip of standardised dimensions. The tip of the cone is placed in contact with the levelled surface of the grease, the cone is allowed to penetrate the grease for five seconds and the depth penetrated in tenths of a millimeter as shown on a graduated dial is the measure of the consistency of the grease. The determination is made with the grease at a temperature of 77° F.

An important point that should be borne in mind is that the original consistency of a grease is determined by a variety of factors which are very difficult to control. The soap content is the most important single factor, but the kind of fats used, the method of manufacture, the final water content, the amount of stirring during manufacture, the temperature at which the grease is run off from the kettle and the rate of cooling are all very important in determining the final consistency. Since it is impossible to maintain each of these factors constant for every batch of grease made, the original consistencies of greases of the same type are bound to vary within certain limits.

Any handling of the grease after it has set or any remelting will greatly change the consistency. The original consistency also changes to some extent on standing—most rapidly during the first 48 hours and only slowly thereafter.

Although many tests are based on the original consistency of a grease, this consistency rarely bears any relationship to the consistency that really matters, *i.e.*, that at the actual point of application of the lubricant to the bearing. At this stage the grease has almost invariably reached its condition of maximum softness.

Consistency tests on the majority of greases should be made therefore on greases that have been reduced by "working" to the condition in which further working fails to reduce the consistency. For this purpose a special grease worker is employed for use when testing with the A.S.T.M. penetrometer.

This grease worker consists of a metal cylinder of standard dimensions fitted with a perforated plunger which is forced through the grease a definite number of times.

The condition of a grease in its original consistency is called the unworked consistency; that in its condition of maximum breakdown. its worked consistency.

All cup greases except the very hardest should be standardized upon their worked consistencies.

On the other hand, for very hard greases used in block form, the original or unworked consistency is generally the property of practical importance and should be the object of the test.

One further point of importance. Greases become softer with rising temperature, but it cannot be assumed that because one grease is softer than another at normal temperatures it will necessarily be softer throughout the whole range of temperatures up to their respective melting-points. One grease may maintain its consistency practically unchanged until it melts, whereas another may soften considerably.

It is of importance, therefore, especially with block greases or others intended for use under high temperature conditions, that the consistency be tested over a range of temperatures.

It is still customary for cup greases to be arbitrarily classified according to certain trade standards—*i.e.*, No. 2 Cup, No. 00 Cup, etc.—but without doubt in future this will decrease in favour of a definite range based upon actual consistency measurements.

2. Melting-Point (or Flow Point).

The melting-point of a grease is one of the controlling factors in grease lubrication. It is quite obvious that if a bearing temperature is 250° F. a grease having a melting-point of only 200° F. should not be used.

On the other hand, the tendency is to attach far too much importance to melting-point. The fact that two greases have the same melting-point does not indicate that they can be used for the same service.

Very few greases have a definite melting-point, but gradually soften as the temperature is raised. This can be demonstrated by determining the consistency of a grease by the A.S.T.M. method over a range of temperatures. The grease will soften as the temperature is raised, until it becomes so soft or semi-fluid that the consistency cannot be determined. In this condition the grease is a viscous liquid rather than a plastic solid.

There have been many methods devised for determining the melting-point of a grease, and it may be said that there is no generally accepted method.

In one method a plug of grease may be drawn up into a capillary tube which is attached by a rubber band to a thermometer. The thermometer and tube are then immersed in a beaker of water, which is heated at a constant rate, and the melting-point is the temperature at which the plug of grease moves up the tube.

A commonly used method is the drop point whereby a small amount of grease is placed upon the bulb of a thermometer and suspended in a test tube. The tube is slowly heated, and the temperature at which the grease drops off the bulb is taken as the melting-point of the product.

Other methods include those in which a steel ball of definite weight is placed upon a definite volume of grease and the temperature noted at which the ball sinks into or through the grease. Again, it is possible to determine the temperature at which a definite weight of mercury forces a plug of grease through a glass tube.

The method used with most satisfaction by the author is that of Ubbelohde. This test is made using a thermometer to which is attached a metal ring supporting a glass cup 10 mm. long with an opening in the bottom 3 mm. in diameter. The cup is filled with grease, taking care to avoid the inclusion of air bubbles. When the cup is inserted in its holder the thermometer bulb rests in the grease. The thermometer is supported in a wide test-tube, which in turn is immersed in a beaker or wider tube containing glycerine, white oil or other suitable medium, and the temperature of this is raised fairly rapidly to within 20° of the anticipated melting-point, then at the rate of 1° per min. The Ubbelohde melting-point is the temperature at which the first drop of molten grease falls from the cup.

The Ubbelohde method will be found of service for all cup greases of melting-point below 100° C., and for the majority of anhydrous greases, including the block greases, with melting-points above this temperature. Greases with high moisture contents are inclined to be blown out of the cup at temperatures near 100° C., and greases containing filling matter are apt to clog the aperture of the cup.

The melting-point of a grease is primarily dependent upon the type and quantity of soap present. For lime soap greases the increase of melting-point for increase of soap content is relatively small, and the majority of such cup greases have Ubbelohde melting-points in the region of 200 to 210° F.

On the other hand, with soda soap greases, a relatively small increase in soap content will normally produce an appreciable increase in melting-point. The usual sponge greases have melting-points within the range of 250–300° F., whilst soda soap greases of the dehydrated block type can be produced with melting-points of 350–400° F.

From what has been said up till now, it will be realised that consistency and melting-point in greases are inter-related functions and that melting-points above everything else depend upon the method by which they are determined. There is no reason why melting-points by one method should bear any relationship whatsoever to those determined in some other way or to the behaviour of a grease in a bearing.

In practice, the greatest value of a melting-point determination is to the grease-maker who adopts one method simply and solely to ensure that he maintains his products at a uniform standard.

3. Stability.

The stability of a grease in storage depends primarily upon the quality of the ingredients used in manufacture and the efficiency of the manufacturing process.

Greases containing poor quality or unstable fats are liable to go rancid on keeping or to oxidise and change colour. The presence of excessive quantities of free fats or free fatty acids in the finished grease are liable to promote this action. On the other hand, with cup greases especially, the presence of a large quantity of free lime will cause separation of the soap from the mineral oil during storage.

Many specialised tests have been devised to determine the stability of greases with respect to definite service requirements. For instance, ball and roller bearing lubricants are usually subjected to a heating or sweating test to ensure that mineral oil separation shall not occur ; also, to a test to make sure that they shall be free from corrosive properties. Greases for use in universal joints may appropriately be subjected to centrifugal action to determine the possibility of oil separation with resultant hardening of the grease.

4. Colour and Odour.

The natural colour of lubricating greases depends entirely upon the colour of the fats and the mineral oils used in their manufacture.

The colour of all fats is liable to slight variations which may alter the depth of colour in the finished grease.

Greases may change colour during storage due to several reasons. Practically all greases mature with ageing, and in doing so they tend to become clearer and more transparent. This results in the colour of the mineral oil becoming more apparent, so that the older matured grease appears a different colour from the freshly made grease.

Oxidation and the drying out of moisture are also responsible for colour changes.

The majority of greases will bleach if kept in strong light, so that a sample that has been retained for some time will often be appreciably lighter in colour than the freshly made grease.

Although colour, to a certain extent, indicates the quality of the constituents of a grease, it is by no means a sure guide. A dark coloured grease may contain highly refined products, yet owing to the temperature of manufacture a darkening of colour will be brought about that is unavoidable.

The majority of fats used in grease making possess a decided odour, and even after saponification they may at times be detected in the resulting grease. For this reason some of the most carefully prepared greases, such as those used in factories handling food stuffs, are slightly scented.

On the other hand, highly scented ordinary general purpose greases should be looked upon with suspicion, as the scent may be there to mask the offensive smell of a rancid fat or a disagreeably smelling oils. Rancid fats should not be used in grease making because of their general instability and rapid deterioration usually resulting in the development of acidity. Untreated oil distillates are also objectionable owing to their instability and tendency towards gum formation.

5. Load Carrying Capacity, etc.

Time will not permit more than the briefest mention of the methods of testing certain other characteristics of grease.

For methods of determining film strength and load carrying capacities of lubricants, those interested must be referred to a paper by C. M. Larson on the Classification of Transmission and Rear-Axle Lubricants presented at the Detroit meeting of the American Society of Automotive Engineers in January, 1931, and to the publications of the Timken Roller Bearing Company (also Automotive Industries, February 14, 1931). The Timken Lubricant Tester comprises a mandrel mounted on two bearings having a tapered portion on its outer end on which a standard Timken bearing cup is held by means of a nut. Directly beneath

the revolving cup a test block ½ in. sq. by ¾ in. long is mounted on a lever supported on a knife edge by a second lever, which is similarly mounted on a universal knife edge on the base of the machine. This arrangement assures that the test block will at all times be parallel to the revolving cup.

The first lever is loaded by means of weights, thus producing a certain pressure per sq. in. of surface area in contact on the square test block. The second lever is a friction lever, and when this is balanced with weights the co-efficient of friction can be determined.

The lubricant is fed by gravity or by a pump over the test block and the tests are run at a uniform speed. The test block is examined at regular periods after increasing the weights to determine at what load scuffing starts.

The Practical Examination of Lubricating Greases.

A complete examination and analysis of a lubricating grease is a lengthy and complicated piece of work. However, it is often necessary to get a general idea of the composition and main characteristics of such a product. In these cases much will depend upon the skill and experience of the analyst, and it will usually pay to make a brief qualitative examination to determine the most appropriate means of approaching the analysis.

It is especially desirable to differentiate between straightforward products made from soap and mineral oils, such as cup greases, where the examination will present no difficulty, and those lubricating greases made from combinations of pitches with mineral oils and perhaps soaps, fats and fillers as well.

The examination of any lubricating grease is composed of two parts :—

(1) Physical.
(2) Chemical.

Under the physical part can be classified the following tests :—

Consistency.
Melting-point.
Heat stability.
Colour and odour.
General characteristics of more or less physical nature.

The chemical part includes :—

Nature and percentage of soap.
Nature and percentage of free fatty matter.
Nature and percentage of mineral oil.
Nature and percentage of filler.
Percentage of water.
Percentage of glycerine.
Free acidity or alkalinity.

Consistency.—The A.S.T.M. Penetrometer is by far the most convenient for control purposes. Measurements should be made on the worked and unworked grease at 77° F. and, according to the type of grease, at several other temperatures.

For lubricants softer than a 400 penetration the MacMichael torsion viscometer or the Saybolt Furol instrument are suitable.

Melting-point.—The melting-point should be determined by a method suitable to the type of grease being examined. The Ubbelohde method will be found to cover the widest range of requirements.

Heat Stability.—Separate portions of the grease should be maintained for several hours at temperatures just below and just above the melting-point of the grease and their condition noted after allowing to cool and placing on one side overnight.

Colour, Odour and General Physical Characteristics.—These are recorded in the usual manner and are more or less subject to the personal observation of the chemist.

Chemical Examination.—It is not proposed in this paper to go into the subject of the analysis of greases, but simply to make one or two observations.

Soap Content.—The type of saponifying base can be determined from the ash of the grease. With lime greases the total lime content can be determined in the ash of the grease.

The combined fatty acids can be isolated by the customary acid splitting method. More conveniently and quickly, the soap can be separated by centrifugal means after diluting with a light petroleum spirit.

By the centrifugal method the soap will retain the moisture, glycerine and fillers, whilst the petroleum spirit extract will hold the mineral oil and free fatty matter.

Mineral Oil.—Sufficient mineral oil should be isolated for a determination of specific gravity, viscosity (at two temperatures at least), flash point, cold test and colour.

Investigation and Development.

There are opportunities for research in every direction in connection with grease, since less attention has been paid to this subject than to any other branch of petroleum technology.

Very little is known regarding the actual lubricating merits of the soaps from different fats, of the influence of the character and the origin of the mineral oil, or of the importance of the presence of free fat, free alkali or acid, water, glycerine and additional materials.

Pressure saponification has made accessible to the grease-maker a large number of fats that previously were neglected owing to the

difficulty of saponification or to the unsatisfactory results obtained when using the older open type steam kettle. Methods of manufacture generally are still capable of improvement, and the use of colloidal or emulsifying mills is well worth study.

Fresh types of soaps and new gelling agents for mineral oils are available, and the whole subject is awaiting investigation from every angle.

It is realised that this paper is incomplete in certain directions, for instance, operating conditions influencing the choice of lubricating greases, methods of application and industrial uses, and troubles that may be experienced through faulty selection or application have received no mention.

An endeavour has been made to cover the subject from an angle of most interest to the Petroleum Technologist—*i.e.*, from the point of view of the types of lubricating greases, their manufacture and their functions as lubricants.

At the outset of this paper the author had in mind suggesting at this stage that the methods of testing lubricating greases should receive some attention from the Standardization Committee of the Institution. It would appear doubtful, however, whether such methods have yet reached the stage where standardization is possible.

In these circumstances the author does feel that the formation of a separate section or group within the Institution where questions relative to lubricating grease matters would be the primary subject for discussion would benefit the Industry as a whole and bring further credit to the Institution of Petroleum Technologists. The views of any other members that might be interested in such a group would be very welcome.

Acknowledgments.

The author desires to express his thanks to the Board of Directors of the Vacuum Oil Company, Ltd., for permission to present this paper, and to his colleagues for their helpful criticism and advice.

He is also indebted to the Sowers Manufacturing Company, of Buffalo, New York, makers of the Dopp kettles; to the British Timken Company, makers of the Timken Lubricant Testing Machine; to Messrs. W. J. Fraser and Company, Ltd., Dagenham; and to John Varley, Ltd., St. Helens; for the loan of photographs. The grease-testing apparatus demonstrated was kindly loaned by Messrs. A. Gallenkamp and Company, Ltd.

DISCUSSION.

The President remarked that engineers always responded to the word "corrosion," geologists to the expression "origin of oil" and chemists to the word "lubrication." In order to make the

fullest use of the short time available for discussion he did not propose to comment on the paper and would call on Prof. Brame to open the discussion.

Prof. J. S. S. Brame thought that Mr. Garlick's address was the first contribution that the Institution had had on the subject, as a separate paper. For that reason it was very welcome. Mr. Garlick said that there was an immense scope for research—"The subject was awaiting investigation from every angle." Everyone would agree with that. Grease had been a Cinderella amongst lubricants. Many very primitive greases had been successfully used for various purposes, and a mental attitude had rather been developed of accepting almost anything as being a suitable grease, as long as it was cheap. Several points occurred to one where research obviously would be valuable. One which particularly struck him was the influence of the glycerin on the constitution and behaviour of the greases. Another one was in connection with the lead soap greases where, it was said, certain of the oils were quite unsuitable for mixing with lead soap ; that in fact the lead soap was insoluble in some of them, and that the effect of the same quantity of the lead soap on different oils (as far as the viscosity of the oil was concerned) was extraordinarily different. That same sort of difference was met with in other cases, namely, in the case of the zinc oxide lubricant to which reference had been made. With certain oils, the addition of a given quantity of zinc oxide would affect the viscosity to no very great degree, but on the other hand it would make another oil—he would not say absolutely set—but so firm that pouring was quite out of the question. The effect, therefore, of these other ingredients on the consistency and so on was of very great importance.

The author suggested towards the very end of the paper the possibility of the formation of a Group in the Institution to deal with greases. The question arose whether there was sufficient material available to keep such a Group busy. The subject was at present very largely empirical, and information and experience in the past had been so often regarded as proprietary material that it was very doubtful whether much would come from it ; but if such a Group could stimulate research on these many problems, and discuss researches, then undoubtedly it would be of very great value.

There was only one other reference he particularly wanted to make, and that was to the standardization question. The author said that the standardization of testing of lubricating greases should receive some attention at the hands of the Institution, and rather doubted whether such methods had yet reached the stage where standardization was possible. The members would be aware

that the Standardization Committee and sub-Committees of the Institution had published standard methods of testing lubricants, with the exception of greases, but they probably did not know that a Panel of the Lubricating Oil Committee had been considering greases, and that certain tests had been recommended to the Committee. They required confirming, but the tests considered including consistency (cone test with the penetrometer), the melting point test, the drop test (the Ubbelohde method), ash, water, acidity and alkalinity. Some considerable progress had already been made in considering this important subject. He would also remind the members that the A.S.T.M. had standardized an analysis of greases (Serial Designation D128–1927). The scheme of analysis had been set forth in detail and had been approved in America. The Penetration Test (D217–33T) at present was only a tentative test. It was not established so firmly that there might not be some modification of it. However, it would be seen that the question had received attention both in America and in this country.

Dr. F. H. Garner thought all must agree that the paper, within the limits which Mr. Garlick had set himself, was very complete. The author was to be congratulated on the thoroughness with which he had dealt with the various methods of manufacture, and particularly on the way in which he had summarised the properties of various types of lubricant. Naturally, in a paper where he was dealing with the manufacture of greases from soda, lime, potash, lead, aluminium, zinc and barium bases, it was not possible for it to be complete, but he thought it would have been an advantage if the members could have had further information on the new types of grease, such as the aluminium soap greases.

Mr. Garlick said " From the colloidal point of view cup greases are emulsions of mineral lubricating oil and water stabilised by lime soap " ; where one had 70 to 90 per cent. of mineral oil and only a small percentage of water the point was raised as to the part played by the water in the stabilising of the mixture of lime, soap and oil. It would be remembered that the author dealt with the manufacture of cup grease by the neutralisation of fatty acids, and stated that this type of grease was quite different in certain properties from the ordinary types of cup greases. The only difference between those two types of grease could be the presence or absence of glycerin ; so that it looked as though, in addition to the three constituents, soap, oil and water, the influence of the glycerin was also of special importance.

With regard to the work of the sub-committee on Grease Testing of the Institution, that had been fairly thoroughly dealt with by

Prof. Brame. That sub-committee had tentatively adopted the use of the penetrometer as in the A.S.T.M. method with the standard cone, but had also made provision as an alternative of a simplified apparatus. The penetrometer with the cone had been adopted by the Customs Department as an instrument for use in solving the very difficult problem of distinguishing between solid and liquid. A full description of the use of the penetrometer would be found in the Customs document dealing with this particular problem. (S.R. & O. No. 695 of 1933.)

In addition to the above remarks it should be stated that it is obviously desirable that the more important method of test for greases should be included in "Standard Methods of Testing Petroleum and Its Products." A number of other important properties of grease in addition to those already mentioned were considered by the Grease Panel, but it was felt that these had not yet been sufficiently studied for methods of standardized test procedure to be adopted.

Mr. E. A. Evans said he had been very pleased to have heard the paper, particularly because there was just a hope that a special section or sub-group of the Institution might be able to be formed to consider greases. Prof. Brame did not think that was possible at the moment, but at any rate Mr. Garlick's paper that evening had been the means of bringing together a number of people who definitely specialised in grease, and if some interest could be stimulated amongst those gentlemen he was sure the Institution would benefit.

He had been going to say a few words about standardization of tests, but that had been so adequately dealt with that he must leave it, except to express the hope that they would not be rushing too quickly into standardization and suddenly find that the British Standards Institution was issuing specifications. He feared there would be much more fun in the formulation of those specifications than there had been in the formulation of oil specifications.

The author was well qualified to speak with some authority on the subject; but owing to the already great length of the paper he had not really had time to stress some of the more recent developments in grease manufacture and its application. Personally, he supposed that the anti-friction bearing makers had to be thanked for quite an appreciable amount of the development which they had stimulated within recent years in grease. They had become very fastidious in their requirements, and their requirements had always been a shade ahead of manufacture; but it did seem a pity that such an excellent body of people spent so much money and time on research and yet were not able to come even to a semblance of agreement amongst themselves as to what lubricant should be

used in ball bearings. It did seem that the time should have arrived when they could have decided amongst themselves what should be used. It did complicate the issue enormously.

Mr. Garlick had referred to the differences in grease, although they were made from the same materials. Anybody who had done any work on grease must have realised that point very forcibly. He always thought that a test which was made in his own laboratory was definitely interesting as an illustration of that fact. They were interested in greases for gear box lubrication, and it was very interesting to watch the action of those greases with a stroboscope. Taking two greases of apparently the same manufacture, the same consistency, it could be seen that one grease would go partly over the pinion and then spread out in fan shape, whereas another one would follow the pinion right over. Why it was he did not know, but it was so ; and if the method of manufacture was very carefully standardizaed, that could be repeated.

He desired to refer to a new departure in greases. The author had referred to the load-carrying capacity. Personally, he thought that perhaps it might be rather safer to refer to that as the film rupture strength. There had been quite a lot of discussion in the American journals about the so-called extreme pressure lubricants. The author had referred to the Timken test. If time had permitted perhaps he would have referred to the Almen test, and also to the new one which had been designed by the Bureau of Standards, the result of whose work had been published in the Journal of the Society of Automotive Engineers. Personally, he had not had any experience with the Timken test, but he had had quite a lot of experience with the Almen machine. Mr. Mougey of the General Motors Research Corporation had to be thanked for the work which he and his colleagues had done in the development of that machine, and it might be interesting just to reflect for a moment on the differences between some of the types of greases. For example, all the soda soap greases which he (Mr. Evans) had examined were all very low in film rupture strength—none of them any better than what one would get with any ordinary oil. But the lime soap greases were definitely better from that point of view. He had also carried his investigations into a number of other soaps, like the lead soaps referred to, but, in whatever way he tried to make the lead soaps, he had not been able to lift the film rupture strength above those of the soda soaps. Therefore, he could not see that the lead soaps had any advantage for extreme pressure lubricants. Some of the heavy metallic soaps definitely showed a marked improvement. It might be asked, what uses are there for extreme pressure greases? They are being used for steering boxes etc.

Before concluding he would like to refer to the question of the manufacture of the lead soaps, because it had been very severely criticised. The ball bearing makers had stated that the lead soaps were definitely bad, because they caused wear of the bearings. He thought the explanation was not that the lead soaps inherently caused wear. If they were made by the action of oleic acid or stearic acid on litharge, then a certain amount of oxide of lead remained in suspension, which was really the cause of the excessive wear.

Dr. W. E. J. Broom said a valuable feature of the paper was that it stressed the general lack of knowledge, or perhaps he should say the lack of published knowledge, of the newer types of greases, or what he would prefer to call artificially-thickened mineral oils. In that respect it was somewhat unfortunate that they had received such scanty treatment by the author. For instance, the loss in viscosity of the aluminium oleate-thickened oils on storage presented some interesting problems. By viscosity he meant the resistance to shear under a set of standard conditions since, as was well known, those bodies did not usually obey the normal laws of viscous flow, higher rates of shear giving lower apparent viscosities. However, the fall in viscosity mentioned, which proceeded more rapidly in daylight than in the dark, could occur (although not usually the case) without any visible sedimentation whatsoever. It would thus seem to be due to some structural change in the manner in which the oil and soap molecules were linked. Perhaps the author could advance an opinion as to the mechanism of this change.

It might be considered that such mixtures were inherently unstable and required a suitable stabilising agent to promote some sort of stability. In that connection the role of the mineral oil must not be overlooked, and possibly the right type of oil might supply the stabiliser needed.

Another interesting problem which was not mentioned in the paper was the change which occurred on heating aluminium stearate-thickened oils. For example, with a light oil of 200 Redwood seconds at 70° F. containing about 3 per cent. of such a compound ; on heating to 75° C., the buttery texture changed to the stringy type—not exactly the same as, but rather characteristic of, the oleate-thickened oils. An increase of temperature to 120° C. or so was accompanied by an increase in viscosity, to such an extent that usually the contents of the vessel would adhere to the stirrer and rotate *en masse* with it. Further heating to higher temperatures then caused a decrease in viscosity.

Unfortunately, time was not available for discussing many other interesting properties of these and other thickeners, but it definitely appeared that a certain amount of fundamental research

was badly needed on the subject, although he expected they might have to look to a University Research department to publish such work.

Mr. J. S. Jackson said that it was by no means an easy task to criticise a paper of the kind presented by Mr. Garlick. He thought that it should be welcomed as a preliminary paper preparing the way for further papers dealing with the theories and individual problems connected with the manufacture and testing of grease. He regarded the paper as a very excellent summary of the general information available on the subject from both the manufacturing and the testing point of view. He felt very strongly that the manufacturing problems with which all were familiar would exist as long as the manufacture of grease was left in the hands of comparatively untrained operatives. Such people hid behind the delightful excuse that grease making was an art and not a science. This attitude was entirely wrong. Grease making was a highly technical operation which must be rigidly controlled along scientific lines. We could not possibly rely upon the foreman who rubbed the product between his fingers and decided whether a good grease had been manufactured. Such a grease maker might be a very good judge of the properties of the product at any given moment, but as he was certainly not gifted with second sight, he could not pretend to foretell what the grease would be like after 24 hours or after a week's storage. The real solution undoubtedly lay in rigid control. Open kettles into which variable quantities of water had to be added at critical moments at the direction of the grease maker were probably the source of many of the present difficulties. He wished that the author had given a little more prominence to the pressure type of apparatus in which the process could be accurately controlled.

The problems to be tackled were, however, very real, but undoubtedly some at least were intimately connected with the variable amounts of added water. The author mentioned a cup grease containing 5 per cent. of water. This water content appeared to be excessively high.

The consistency was obviously a very important and very critical feature of any grease. It was a property which the user judged, and judged surprisingly accurately, by the simple process of pushing his finger into the grease. The amazing feature about this apparently crude procedure was that it constituted a very delicate and discriminating test. Unfortunately, the available standard tests for measuring consistency all had distant limitations. In the case of the A.S.T.M. Penetration Test, for instance, it had been found possible to prepare a series of grease samples which, while showing the same A.S.T.M. Penetration, obviously varied

widely in consistency as judged by the sense of touch. Such fictitious results possibly arose from the fact that in accordance with the A.S.T.M. Method the grease was subjected to shock when the cone fell on to the surface. In feeling the texture, the finger was usually inserted gently and moved steadily through the grease. He remarked that the Abraham Consistometer was an attractive apparatus from this point of view since the plunger was forced steadily into the sample at a constant rate. He did not suggest that this Abraham test was entirely satisfactory, but it was a test worthy of consideration.

In view of the colloidal structure and plastic nature, it seemed inevitable that satisfactory methods of test would have to be comparatively elaborate. Attempts had been made to determine the consistency of grease by forcing the product through capillaries under relatively high pressure, but here again the results were still rather puzzling.

He hoped that future papers would deal more fully with the extreme pressure lubricants which had recently attracted a good deal of attention. The Timken tester which had been mentioned had certainly produced very interesting results. The effect of sulphur, either free or combined, was, for instance, quite striking. The author was to be congratulated on preparing the way for a useful series of papers on a very interesting technical problem.

Dr. E. R. Redgrove agreed with the last speaker that the paper, giving much valuable information as it did, could be regarded only as an introduction.

Dealing with the consistency of greases, it had to be remembered that they were very complicated solutions of colloidal substances in a more or less inert body, and he thought a lot depended upon the odd 1 or 2 per cent. of moisture ; in many greases a highly hydroscopic subject, glycerin, was present, and it was therefore quite feasible that atmospheric humidity might affect consistency ; in fact, this influence had been noticed. He thought that more use should be made of the microscope as a means of determining those conditions which influenced the consistency and stability of greases, and he had hoped that the author, in his paper, would have dealt with that aspect of the subject, but no doubt Mr. Garlick would favour the members with another paper at a later date.

Mr. S. E. Bowrey remarked that the testing of consistency over a range of temperatures was more instructive than the determination of melting point as a means of ascertaining the suitability of a grease for a given purpose. The B.S.I. were investigating the possibility of standardizing a simple type of pressure viscometer for use in measuring the consistency of numerous classes of material,

including oils, greases, emulsions, suspensions such as paints, and colloids such as glue. Such an instrument might have advantages over the penetrometer for plotting consistency-temperature curves.

The need, already voiced, for better knowledge of the requirements of the user in regard to greases was urgent. It would be interesting if at some future meeting the ball-bearing experts could be induced to take part in the discussion and help to settle what are the essentials of the ideal ball-bearing grease for various applications. The author spoke of the pressure kettle as a modern device in grease manufacture. He wondered whether this description was justified. His own firm's original pressure plant was designed and put into use in 1917, and he would be interested to know whether it was the first.

Mr. C. I. Kelly said there was one other point which he thought the author might deal with more fully in his reply to the discussion, and that was the stability of greases in storage, especially from the viewpoint of the bleeding of oil out of the grease. If the author has done any work in that direction the members no doubt would be interested to know what were the results. He would draw the attention of members to the paper by Mr. Herschel,* in which work on this subject, among others, is reported.

Mr. Jackson had referred to the inclination of grease buyers to put a finger into samples to see what the consistency was like. It would be informative to know whether the user would test a tin of grease in this empirical way if it had a thin layer of separated oil on the top of its contents. Personally, he did not think the user would ; he would discard it immediately. Considerations such as these prompted his question regarding grease stability at ambient temperatures.

In reference to the question of aluminium soap greases, would the author tell us if he had ever been so fortunate as to have met such commodities as aluminium stearate or aluminium oleate in his long experience ; he expected the author to confirm the fact that persons manufacturing or buying aluminium soaps were taking too much for granted in believing they were making or procuring " aluminium stearate " or " aluminium oleate " and nothing else ?

Mr. G. H. Thornley remarked that the author had referred to the great stability of greases made by the cold process from fatty acids and lime, and to the fact that they could be heated to a temperature of 250° F. and on cooling retain their original characteristics and transparency. In his own experience that had always

* A Procedure for Measuring Bleeding of Cup Greases, *Proc. A.S.T.M.*, 1933, **33**, Pt. I., 343-347.

been due to a proportion of free fatty acid ; if the grease was neutral, whether it was made in the first place from free fatty acid or neutral fats, its heat-stability was not so great as it was if it contained 1 or 2 per cent. of free fatty acids.

Mr. H. G. Catterall asked the author just exactly what he meant by "hair greases"—their merits and demerits, as distinct from wool sponge greases, and whether they were marketed in Great Britain.

Mr. H. S. Garlick, in reply, said that Prof. Brame, Dr. Garner and Dr. Redgrove had referred to the influence of glycerin in greases. Without doubt, glycerin had a very important effect. The presence or absence of glycerin appreciably altered the structure and length and strength of the fibres in a sponge grease. Glycerin was present in a cup grease formed by saponifying a neutral fat with lime, yet such a grease did not behave in an identical manner to a similar grease formed by neutralising the fatty acids derived from the same type of fat to which glycerin had been added. The effect of glycerin as a stabilising agent was a problem well worthy of study.

The author agrees with Mr. Evans that ball and roller bearing manufacturers have influenced certain phases of grease manufacture to a great extent and without doubt it has been due to their efforts and insistance that stable cup greases of very low moisture content are available to-day, and that specialised types of lubricants have been developed. As Mr. Bowrey states, it is to be regretted that these people could not get together amongst themselves, and with the grease manufacturers, in an endeavour to obtain agreement as to the most suitable universal lubricant for the various types of anti-friction bearings, thereby reducing the number of types at present recommended.

The author's experience with the various types of soaps in extreme pressure lubricants would not seem to be quite the same as that of Mr. Evans, and some further particulars on this subject are undoubtedly wanted. A lot of the trouble experienced with lead-base lubricants, either from separation or undue bearing wear, was due to the choice of unsuitable materials or to faulty manufacture, and in this latter connection manufacture in a pressure kettle could help a great deal.

Dr. Broom had referred to the somewhat scanty treatment by the author of the newer types of greases. As already pointed out, it has not been the author's intention in this paper to deal exhaustively with the subject at all. A great deal of work has been going on relative to new types of greases, and undoubtedly a lot of knowledge has been accumulated which manufacturers are

hesitant about publishing. It is here that the value of a group within the Institution, as suggested by the author, should be manifest, as it should function as a clearing-house for such information, where problems could be discussed to mutual advantage.

In the author's opinion the reason for the perplexing changes and variation in properties of mineral oil solutions of aluminium soaps is to be found in the chemical composition and purity of the aluminium soaps used. Commercial grades of aluminium stearate and aluminium oleate from different manufacturers give widely different results, whether from a viscosity or a stability consideration, and the only way such problems can be tackled is by starting with definitely standardized and known raw materials.

Replying to Mr. Kelly, there is no doubt but that it would be definitely advantageous if similar types of grease could be grouped according to definite consistency standards and thus do away with the present often ambiguous trade classifications. This is a matter the grease panel of the Standardization Committee might well consider.

Gravity Measurements in the Great Hungarian Plain.

By H. de Boeckh.

[In 1931 the late Dr. Hugo de Boeckh, Director of the Geological Survey of Hungary, delivered a series of advanced lectures in the University of London, entitled "Regional Geology and Tectonics." As Dr. de Boeckh had been closely associated with the pioneer geophysical investigations of Baron Eötvös, he was asked to introduce into his lectures an account of early work with the torsion balance. Dr. de Boeckh's lectures were greatly appreciated, and on his return to Hungary he began to prepare them for publication. Unfortunately, he died suddenly in the autumn of 1931, shortly after he had conducted me over the ground now rendered classical by the early torsion balance work. His widow, Mme. de Boeckh, placed the manuscript in my hands to deal with as seemed most appropriate. The following paper formed part of the manuscript. It is in the nature of a historical document, and Dr. de Boeckh's phraseology has been adhered to as closely as possible.—P. G. H. Boswell.]

In the Great Hungarian Plain the surface evidence does not tell us much about the underground rocks. From the study of the gravel deposits south of the Bakony, Lewis de Lóczy, Senior, came to the conclusion that there must have been dry land to the south-east of the Bakony mountains. Later on, he expressed the view that the whole of the Great Plain was a land area from the end of the Cretaceous Period until the Pliocene, and was a continuation of the Rhodope mass; in fact, he suspected that below the Great Plain were remnants of the Altaids or Variscan mountain ranges.

The Plain extends over miles and miles of absolutely level country. When I was faced in 1913 with the problem of the Great Plain in connection with exploration work, I realised that the geologist must look for help to the geophysicist.

At this time Baron Roland Eötvös had constructed a very sensitive instrument, the torsion balance, with which he was carrying out measurements to determine the surface curvature of the earth.

Since 1891 he had been making observations at several places, and from 1906 onwards he obtained a yearly subsidy from the Hungarian Government. In 1912 and 1913 he worked in Transylvania. As a result of his measurements he constructed a gravity profile.

When studying this profile and examining his charts, which showed the distribution of gravity, I realised the geological significance of his measurements, for I observed that his minima coincided with anticlines and his maxima with synclines. In the Transylvanian basin salt appears in many anticlines and may be supposed to occur in other anticlines also.

In the simple case where folded rocks occur below a level surface, and taking into account the fact that the older rocks are generally

more consolidated than the younger rocks, we see that above the axial part of an anticline greater gravity must be expected than above a syncline. The same reasoning holds good even in hilly country, but here the so-called " terrain effect " makes the measurements difficult or even impossible.

If, in the case of anticlines, a less dense rock, *e.g.*, salt, is present, the picture will be inverted and the minimum of gravity will lie above the anticline. It was further evident that as the gravity is inversely proportional to the distance of the acting masses, the masses closer to the surface must exert a greater influence upon the instruments than those which are situated far away. If, for example, we put in the corners of a room three heaps of iron and place the instrument close to these masses, making a tight net of observations, the masses of the different heaps can be exactly determined by means of the instrumental observations.

If the instrument is lifted to a level at which its distance is great as compared with the distance of the iron masses from one another, only resultants are obtained. If the results are plotted, the curve of gravity will be very gentle. It will be the same in the case of the earth, for the deep-seated masses will give resultants and a very gentle curve of gravity change. The rocks nearer to the surface will superpose a strong effect on this curve.

The more deep-rooted the rocks, the more gentle will be the curve of gravity given by them and we can say that, so far as the instrument is concerned, the gravity will be compensated at a certain depth—a statement which I can strongly recommend to those interested in isostasy.

The conclusion may thus be drawn that the results given by the torsion balance must give information about rocks if they are not situated at too great a depth.

But there is a further consideration. In the case of an anticline or syncline there will always be, on the sides of every section, two points where the gravity will be the same. With concordant folding these points will correspond to the same thickness of rocks if a thick sedimentary series is present. If a tight net of observations is made and the points with the same gravity are connected, the isogammae, or lines of equal gravity, will represent something like an underground contour map. Indeed, when I studied the isogammae of the surroundings of Kecskemét as published by Baron Eötvös, the resemblance of the isogammae to underground contour-lines was obvious (Fig. 1).

Baron Eötvös realised the difference between the forms shown by the isogammae and the actual surface forms of the earth. In the paper which he published in 1912, on the measurements carried out between 1909 and 1911, he referred to the features depicted by

FIG. 1.

SUBTERRANEAN ANOMALIES OF GRAVITY IN THE GREAT HUNGARIAN PLAIN.

his measurements as follows : " Whereas, on the surface, mountain ranges chiefly catch our eye, which ranges are arranged in trends and often have very ragged outlines, the torsion balance enables us to recognise accumulations of masses at depth ; these masses show more rounded forms which extend over large areas with gentle rising and falling slopes. They are forms like those which can be found at the bottom of the sea and which show some likeness to the stronger features of the Moon. We may, perhaps, see in these the primary forms of the crust of the Earth, which, being protected from the influence of the water and air, have been preserved unchanged during the passing of geological ages."

In the same paper he dealt with the problem as to whether it would be possible to make practical use of his method of observation and said characteristically : " No, we do not wish to do this, in any case not at present, because we have as yet only preliminary information."

He remarked, however, in connection with the great quantities of natural gas which my collaborators and I found in Transylvania, that, according to the geologists, the big accumulations of natural gas are found in the immediate proximity of anticlines. He then noted that the torsion balance could, perhaps, give guidance in finding the anticlines of the Alföld, and the instrumental results should be taken into account.

The profile constructed by Baron Eötvös across Transylvania shows quite distinctly the difference between the views of the distinguished geophysicist and my own interpretation.

Realising the true geological significance of the torsion balance measurements, I asked Baron Eötvös to make measurements at Egbell (Fig. 2), for I had located a well there in 1913, which struck oil early in 1914. If my reasoning about the geological interpretation of the torsion balance measurements was correct, the measurement at Egbell should give a maximum. So I asked Baron Eötvös to have measurements made here also. The survey was carried out in 1915 and 1916, and showed a maximum on the anticline at Egbell.

The Czech geologists have recently postulated a number of faults in the Egbell structure, an assumption which it would be worth while to check by a gravity survey, for the torsion balance measurements can be used to determine faults as well as folds.

In 1917, I requested Baron Eötvös to have regular surveys made in the Great Hungarian Plain. These measurements have since been carried out, and were continued after I had left Government service in 1921.

According to my interpretation the maxima would mean elevations where the rocks are closer to the surface, and the minima

EGBELL.

1:300.000

FIG. 2.

GRAVITY ANOMALIES: EGBELL AREA, HUNGARY.

synclines. To decide this, I determined during the war to have two boreholes drilled, one above a maximum and one above a minimum. If my reasoning was correct, the borehole situated on an anticline must reach older rocks sooner than the borehole in a syncline.

I had selected as sites the maximum near Hajduszoboszlo (Fig. 3) and the minimum to the N.N.W. of it. Unfortunately, the two boreholes could not be begun at once and the second borehole above the maximum has been stopped at a depth of 300 metres and moved 4 km. to the West, down the flank of the maximum.

Here one borehole has been carried down to a depth of 2023 metres and has encountered pebbles and conglomerates below the Pontian, at a depth of 1425 metres, and rocks which are either of lower Permian or of Carboniferous age at 1465 metres.

One borehole at Debreczen reached, at a depth of 1465 metres, red sandstones and grits of perhaps Permian or Mesozoic age. But most probably they represent a conglomerate composed of those old rocks. Above these sandstones and grits, Sarmatian rocks were encountered at 1318 metres in depth, and were found to contain many dacite tuff beds.

In my paper, "Contribution to the Stratigraphy and Tectonics of the Iranian Ranges,"* I had expressed the view that, in the Hungarian Basin, Tertiary volcanic material should be present. The borehole of Debreczen has given the evidence and confirmed my prophecy.

From the above it follows that whereas the marginal depressions of the Hungarian Median Mass were formed in early Tertiary times, parts of the central area underwent subsidence only at a later date; hence, we find Sarmatian and Pliocene deposits transgressing older rocks.

The structure of the Great Hungarian Plain is a buried hill structure. Between the maxima are large areas where further work is needed to show what kind of deposits are to be found in the troughs. These areas of synclinal character formed depressions in ancient times and may contain Mesozoic and Nummulitic rocks.

In more recent times anticlinal trends were developed in the central parts of the Great Plain. Evidence has been afforded by shallow pits. These sediments, which are of early Holocene age, are very lenticular; consequently, the dips collected in such deposits are by no means reliable. In the north-eastern marginal depressions, however, structures have been determined in Pleistocene deposits.

* "The Structure of Asia." Edited by J. W. Gregory. Methuen & Co., Ltd., London, 1929.

From the above it is clear that the torsion balance measurements have shown that the foundation of the Great Plain is an irregular surface. The isogammae are the expression of a "buried hill" structure.

FIG. 3.

GRAVITY ANOMALIES : DEBRECZEN AREA, HUNGARY.

The depth of the old rocks seems to be 1500 metres. Baron Eötvös had calculated the depth of the old rocks (which, according to him, were perhaps the first crust of the earth) as about 100 metres deep at Kecskemét, but he added that this figure would hold good only if the specific gravity of the deeper rocks were 2·6, and that of the surface rocks 2·0. If intermediate values occurred, the depth must be greater than that mentioned.

The ancient rocks that were encountered and the conglomerates found above them, contain rocks entirely similar to the Palæozoics of the Bükk and of the Bihar, so that L. de Lóczy, Senior, was right in his conjecture that Altaids would be found at the bottom of the Alföld.

Some Observations on the Flow of Oil into Oil Wells.*

By S. F. Shaw, M.A.I.M.E., M.A.S.M.E. (Member).

The flow of oil into a well is caused by a higher pressure existing in the reservoir than in the well. This pressure may exist because of there being a mixture of gas with the oil either as free gas or as dissolved gas, or because there is a water head behind the oil. Also, if the oil formation were compressible, it is conceivable that the oil could be partly squeezed out of the formation into the well by the weight of the overlying rocks settling down and compacting the oil-reservoir rock.

The oil cannot continue to flow into the well unless it is removed from the well; it cannot flow into the well more rapidly than it is removed from the well. For a limited time, however, until the well is emptied to the bottom, the oil could be removed at a greater rate than that at which it enters the well.

There may be sufficient water pressure behind the oil to force it out of the sand into the well and up to the surface, similar to an artesian water well. Also, if there be sufficient gas present, the oil may be propelled through the sand to the well and lifted to the surface, even though the pressure be less than that corresponding to the weight of a solid column of oil stretching from the bottom to the top of the well.

Oil will flow more readily through a very open porous medium than through a very tight sand. It will flow more readily if it is thin, like gasoline, than if it be thick, like molasses. It will flow more readily if there be a high pressure behind the oil than if this pressure be low. Lastly, it will flow more readily if the pressure at the bottom of the well is held at a low point than if the pressure is maintained at a high point. The pressure at the bottom of the well is governed by the rate at which the oil is removed from the well; that is, if the oil be produced at a high rate, it maintains the level of the oil at a lower point.

The curve in Fig. 1 illustrates the relation between the flow of oil into a well and the pressure maintained at the bottom of the well. This pressure is controlled by the rate at which the oil is removed from the well, irrespective of the method used for lifting the oil, whether it be natural flow, gas-lift flow, swabbing, pumping or any other means.[1,2,3]

In Fig. 1 it will be noted that, as the fluid level is lowered, there is an increase in the production, but the rate of increase in production is not proportional to the rate at which the fluid level

* Paper received October 13, 1933.

is lowered. In the example, the average daily rate of production is 10 barrels per foot for every foot that the oil level is lowered from 250 ft. to 150 ft., but this rate is increased only 100 barrels per day or 1 barrel per ft. when lowered between the levels of 150 ft. and 50 ft., making an average rate of $5\frac{1}{2}$ barrels per ft. that the oil level is lowered between 250 and 50 ft.

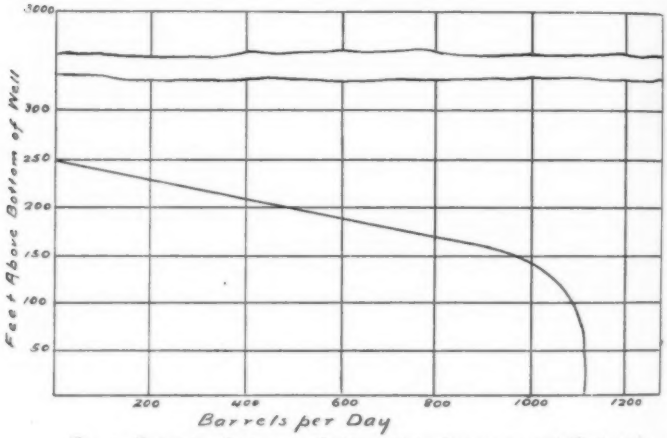

Fig. 1. Relation Between Bottom Hole Pressure and Capacity.

During the early life of many wells, in fact in most wells in deep fields, the oil flows naturally to the surface, by reason of the high pressure in the reservoir and the comparatively large quantity of gas contained in the oil. After the pressure declines sufficiently, and after a certain proportion of the gas has been produced with the oil, these wells usually refuse to flow naturally, and means must be provided for lifting the oil out of the well.

After the well has ceased to flow naturally, if the quantity to be produced is large, or if the depth of the well is great, the gas-lift must be employed, as it is the only method that so far has been found satisfactory under these conditions. The gas-lift can be employed also in shallow wells to handle either large or small production, so also can the swab or pump be employed in shallow wells for small or large production and for medium production in wells of medium depth.

In handling wells on gas-lift, some very interesting observations have been made that furnish data indicative as to how the wells can be produced to best advantage. The testing of large wells

produced by gas-lift at regular intervals has been the means of making a large profit by companies handling their production in this manner.

These tests are made at fairly regular intervals and consist in admitting gas at different rates, and at each rate making observations of the quantity of oil produced, the quantity of input and output gas, and the bottom-hole pressure. From these data, curves are plotted which indicate the best rate at which to produce the well, depending on the object to be achieved as to whether the maximum production is desired or the maximum efficiency. From the observations so made there are calculated the input and output gas per barrel of oil together with the gas-oil ratio. These data are often found to indicate that changes in the sizes of the tubing would be beneficial.

Illustration of Tests Made on Flowing Wells.

Tests illustrating the points mentioned in the foregoing paragraphs will be given in examples that follow. A well of approximately 4000 ft. in depth was tested with the results given in Table I :

Table I.

Data observed when Testing a Gas-lift Well 4000 ft. in Depth.

Test No.	Cu. Ft. Gas per Min. Input.	Output.	Barrels Oil per Day.	Cu. Ft. Gas per Barrel Oil. Input.	Output.	G.O. Ratio.
1	749	1025	978	1105	1512	407
2	1055	1475	1326	1134	1600	466
3	1882	2431	1480	1831	2365	534
4	1835	2333	1498	1764	2243	479
5	2030	2580	1458	2003	2545	542
6	2280	2780	1386	2369	2885	516
7	2543	3065	1260	2906	3503	597

The production in barrels per day and the output factor per barrel are plotted in Fig. 2. It will be noted that the output factor per barrel gradually increases as the number of barrels per day is increased. This is due to the fact that, as the productive rate is increased, the pressure at the bottom of the hole is reduced, in consequence of which the pressure of the gas is reduced at the lower end of the tubing, resulting in a reduced amount of energy contained in the gas. Coincident with this reduction in the energy available is the fact that the distance that the oil must be lifted has been increased. These two factors make necessary an increase in the number of cu. ft. of gas required to lift a barrel of the oil. The highest lifting efficiency in this well was found when producing at the rate of 978 barrels per day, at which rate the quantity of output gas was 1512 cu. ft. per barrel. This point of highest lifting efficiency is found by drawing a tangent to the curve at

point *b* from the point of origin of the co-ordinates.[4, 5, 6] Further increase of gas reaches the point of maximum production, as shown on the curve by the letter *c*. Still further increase in the quantity of gas used results in reducing the production, as shown by the curve gradually turning backward from *c* toward *d*, and if there were sufficient gas available the curve could finally be carried to the point where there would be no production of oil such as indicated by some point as *d*.

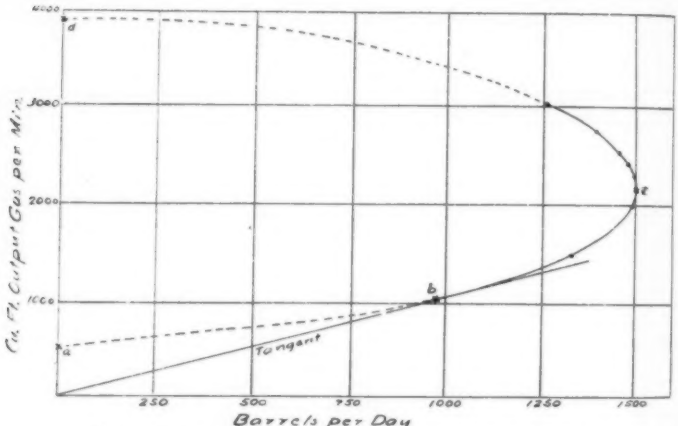

Fig 2. Relation Between Output Gas and Oil Lifted.

The character of the flow at point *b* was very steady, and without a test being made no one would know that the well possessed a higher productive capacity. Nevertheless, it was found by making tests that the production could be increased from 978 barrels per day to 1458 barrels, an increase of nearly 500 barrels per day. The well was also flowing steadily when the output gas amounted to 3065 cu. ft. per min. and production was 1260 barrels per day, and it could easily happen that this excessive quantity of gas was being consumed without any one being aware of the fact that production was being reduced unless such tests were made. When oil commands a high price, it is easy to see where the operator can make a large difference in his monthly returns by producing his wells at the rate that will result in obtaining the maximum advantage.

The injection of a large quantity of gas sets up a high friction loss in the flow pipe, and it requires a greater pressure to overcome

this loss. This higher pressure required at the bottom of the flow pipe is, of course, exerted against the sand, and tends to hold back the oil from coming into the well as freely as if this pressure were reduced.

Another test is given which indicates the advantage of testing a well through two different strings of pipe and comparing the results obtained. The data obtained were not as satisfactory as could have been desired, due to fluctuations caused by the lack of exact control of the gas admitted to the well from the compressor plant, also to lack of exact control over the pressure in the vacuum line to the gasoline plant.

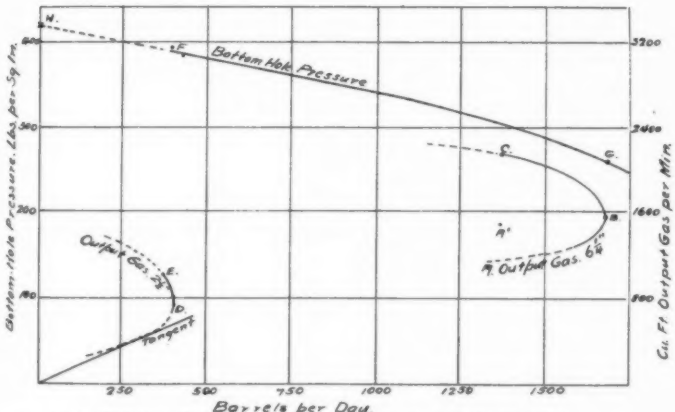

Fig 3. Relation Between Production, Pressure, and Output Gas.

The well was approximately 6000 ft. in depth, cased with 6¼-in. pipe, and had 2½-in. tubing suspended close to the bottom of the well. Data from tests taken while operating this well are given in Table II (see Fig. 3):

TABLE II.

Data on Gas-Lift Operations of a 6000-ft. Well with 6½-in. Casing.

Test No.	Cu. Ft. Gas per Min. Input.	Cu. Ft. Gas per Min. Output.	Barrels Oil per Day.	Cu. Ft. Gas per Barrel Oil. Input.	Cu. Ft. Gas per Barrel Oil. Output.	Cu. Ft. Gas per Barrel Oil. G.O. Ratio.
Through 6¼-in.—						
1	?	1515	1336	?	1635	?
2	868	1554	1676	746	1335	589
3	?	2160	1336	?	2330	?
Through 2½-in.—						
4	?	724	419	?	2450	?
5	?	897	419	?	3080	?
6	?	1035	358	?	4150	?

In Table II it will be noted that the use of the $6\frac{1}{4}$-in. flow string resulted in a much higher production than was possible to obtain through the $2\frac{1}{2}$-in. tubing. The maximum productive rate at the time of the test through the $6\frac{1}{4}$-in. was 1676 barrels (Curve ABC, Fig. 3), while the maximum rate through the $2\frac{1}{2}$-in. was 419 barrels per day (Curve DE).

The bottom-hole flowing pressure, while producing at the maximum rate through the $6\frac{1}{4}$-in., was in the neighbourhood of 260 lb. (Point G), while the flowing pressure when producing through the $2\frac{1}{2}$-in. tubing was approximately 390 lb. (Point E). The pressure-production curve HFG would seem to indicate that with a back pressure on the sand of about 420 lb. the well would cease to produce. It would also lead to the belief that, had the casing been larger than $6\frac{1}{4}$ in., the production would have been increased considerably above 1676 barrels per day. However, with the casing having once been set, it was impracticable to make any change of this kind.

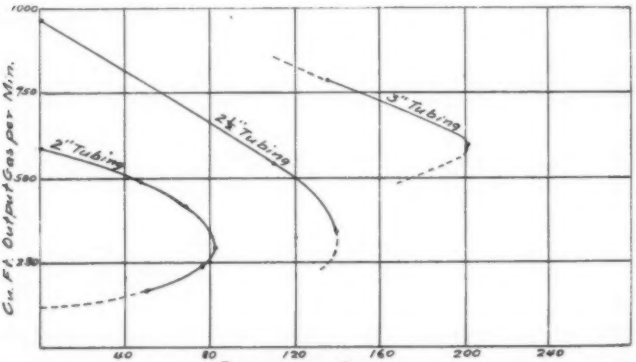

Fig 4. Relation between Production and Output Gas With Different Sizes of Tubing.

Tests of this nature permit the operator to obtain a knowledge concerning his well which could be got in no other way. The maximum productive possibilities are established, the best diameter of pipe can be determined, the quantity of gas necessary to employ can be ascertained, and bottom-hole pressures can be determined.

In another well with depth of about 1800 ft., it was desired to know the best size of tubing to employ, and the quantity of gas that would be required to lift the oil. Accordingly, tests were made with strings of 2-in., $2\frac{1}{2}$-in. and 3-in. in successive order, the data from which are shown in Fig. 4.

It will be observed that the maximum rate possible to obtain through the 2-in. tubing was 84 barrels per day and the quantity of gas per min. was 300 cu. ft. With $2\frac{1}{2}$-in. tubing the maximum production was 130 barrels per day, and the quantity of output gas required was 350 cu. ft. per min.; and with 3-in. tubing the maximum production was 200 barrels per day and the quantity of output gas was 600 cu. ft. per min. It is clearly shown in Fig. 4 that the 3-in. tubing was required to obtain the maximum production.

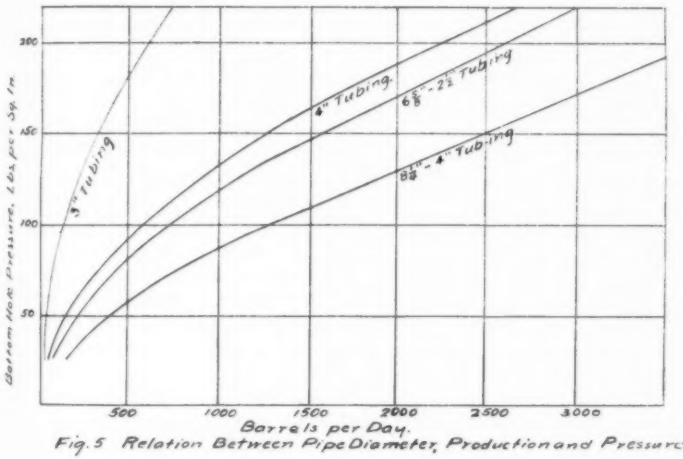

Fig. 5 Relation Between Pipe Diameter, Production and Pressure

The relation between the quantity of oil that can be produced through flow pipe of a given diameter, 4000 ft. in length with given bottom-hole pressure, is shown in Fig. 5.* It will be noted, for example, that with a bottom-hole flowing pressure of 200 lb. there can be 600 barrels per day produced through the 3-in. tubing, 2200 barrels per day through 4-in. tubing, 260 barrels per day through $6\frac{5}{8}$-in. casing and 3750 barrels per day through the $8\frac{1}{4}$-in. casing. These figures apply to conditions similar to those in the Seminole field; for other fields and for other conditions the positions of the curves would be changed.

Similar curves and similar analyses can be made for wells in other oil fields, and it is probable that the operators of such wells would be considerably surprised at the betterment in operations

* S. F. Shaw, "Comparative Data on Bottom-Hole Pressures and Diameter of Flow Strings in Seminole Pool," *Oil & Gas J.*, June 8, 1933, p. 20.

that would result from such regular studies, particularly in the early life of the wells. After wells have declined to a small production, and after equipment has been provided for handling the wells in a certain manner, it is not so easy to work out studies that would justify the operator in changing the method because of the expense involved in installing new equipment.

Gas-Oil Ratios.

The subject of gas-oil ratios is one that has been discussed in various ways. The present discussion is not one touching on conservation, but on that of lifting of oil and its relation to gas-oil ratios.

Any flowing oil well, whether flowing naturally or on gas-lift, is dependent on the lifting power of the gas that comes out of the well with the oil. The oil and gas at the bottom of the well are under higher pressure than at the top of the well. As the gas ascends in the well the pressure becomes gradually reduced and the gas expands. The work done by the gas in expanding from the pressure at the bottom of the hole to the pressure at the top of the pipe provides the energy necessary to lift the oil.

There is a definite quantity of energy in 1 cu. ft. of gas when it expands from a given pressure to a lower pressure, and the maximum quantity of work that can be done by the gas in lifting oil is fixed by the quantity of energy contained in the gas. If there is not sufficient gas present to furnish the energy necessary to lift the oil, the flowing conditions will cease. Table III gives the quantity of gas under a given bottom-hole pressure necessary to lift 1 barrel of oil with gravity of 41° through a pipe of given length where there is no back pressure at the top.

Table III.

Quantity of Gas required to Lift 1 Barrel of 41° Oil.

Bottom-hole Flowing Pressure.	Cu. Ft. of Output Gas required per Barrel. 3500 ft.	5500 ft.	7500 ft.
2000	0	0	85
1000	40	170	400
500	200	600	1250
200	930	2100	3900
100	2100	4300	9500

If the quantity of gas that comes to the well with the oil is sufficiently great, the oil will flow naturally. If this quantity is not sufficient, the deficiency must be made up by gas supplied by a compressor plant or by another gas well. If the well flows naturally the quantity of gas in cu. ft. divided by the number of barrels of oil lifted is termed the gas-oil ratio, although this ratio may not be the same as that which exists in the reservoir, since

some of the gas that comes to the well may be that which escapes from oil remaining in the reservoir.

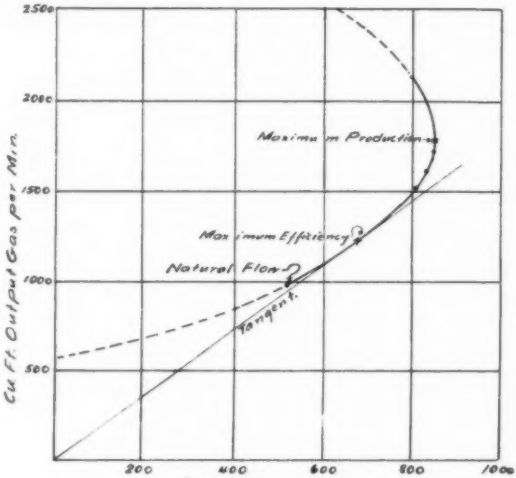

Fig 6. Relation Between Production and Output Gas, in a 4000-Foot Well.

The following example is given to illustrate the relation between the oil lifted, the gas-lift factor, and the gas-oil ratio in a 4000 ft. well, which was tested at different rates. The rate of oil produced was controlled by the quantity of gas admitted to the well from the compressor plant. This quantity of gas was changed at intervals in order to observe the effects of the change (see Fig. 6). The data given in Table IV illustrate one such series of tests.

TABLE IV.

Data on Well produced by Gas-Lift and by Natural Flow.

Test No.	Cu. Ft. Gas per Min. Input.	Cu. Ft. Gas per Min. Output.	Barrels Oil per Day.	Cu. Ft. Gas per Barrel Oil. Input.	Cu. Ft. Gas per Barrel Oil. Output.	Cu. Ft. Gas per Barrel Oil. G.O. Ratio.
1	1018	2153	787	1865	3945	2080
2	777	1911	825	1355	3335	1980
3	597	1718	856	1006	2890	1884
4	507	1623	838	870	2790	1920
5	421	1535	811	747	2725	1978
6	0	973	555	0	2520	2520

It will be observed in Table IV that as the quantity of input gas per minute is reduced, the quantity of oil lifted is also reduced after a certain point is reached. However, the admission of 1018 cu. ft. per min. resulted in a reduction of oil amounting to

about 70 barrels per day from that obtained when admitting 597 cu. ft. per min. This well would flow naturally at the rate of 555 barrels per day, or a rate that is lower than that possible to obtain when gas is admitted to the well from the compressors. The test was carried to the point where all input gas was shut off from the well, at which time it will be noted that although the quantity of gas required to lift a barrel of oil is less than that when gas is admitted from the compressors, nevertheless the gas-oil ratio is higher. When the gas from the compressors is also employed, part of the gas making up the gas-oil ratio is supplied from the compressors. This experience of reducing the gas-oil ratio by means of producing at a higher rate on gas-lift has been the usual experience of the author in most wells where direct comparison could be made.

References.

[1] J. T. Hayward, "Effects of Rate of Production and Producing Equipment upon Gas-Oil Ratios," *Trans. A.M.I.E.*, Pet. Dev. and Tech., 1932, p. 367.

[2] H. N. Marsh and B. H. Robinson, "Means of Controlling Gas-oil Ratios," *Trans. A.M.I.E.*, Pet. Dev. and Tech., 1928–1929, p. 183.

[3] S. F. Shaw, Paper read before Tulsa Section, A.P.I., November, 1928, "Fluid Levels, Back Pressures, and Submergence in Oil Wells."

[4] H. N. Marsh, "A Review of Developments in Gas-Lift and Pumping," World Petroleum Congress, London, 1933, **1**, 493.

[5] S. F. Shaw, "Increasing the Ultimate Recovery of Oil," *Trans. A.I.M.E.*, Pet. Dev. and Tech., 1931, p. 178.

[6] S. F. Shaw, "Unwatering the Tiro General Mine by Air-Lift," *Trans. A.I.M.E.*, Vol. LXIII (1920), p. 429.

A Theoretical Comparison of Petroleum Distillation Systems.

By P. Meyer.*

DISCUSSION.

Mr. P. J. Roelfsema writes as follows: This paper discusses the application of an " overlap coefficient " as a basis for comparing the results of distillation systems. The definition of this coefficient is not simple. *Overlap* is defined as the volume of material in the distillate from a certain quantity of feed which boils above the *cut-point temperature*. Similarly, *underlap* is defined as the quantity of material in the residue which boils below the cut-point temperature. These volumes are stated to be equal. The *cut-point temperature* is defined as the average boiling point of the overlap-underlap, and further as " the temperature at which the same percentage overhead is obtainable by standard column distillation." This overlap volume becomes the *overlap coefficient* if the volume of feed is such as to give a distillation curve of 1 ml. per 1° C., or it can be calculated by dividing the overlap volume per 100 ml. feed by the slope of the standard column curve (in per cent. per degree C.) in the cut-point region.

An equation is developed on the basis of two components and applied to flash vaporisation of a hypothetical mixture having a straight-line distillation curve; this gave a value for the overlap coefficient of 28·5. Data on flash vaporisation of crudes gave values ranging from 13·7 to 27·1, but mostly falling in the range 22–27. This portion of the discussion (dealing with flash vaporisation) seems logical, and may be of value in computing flash distillation overlaps in cases where it is not convenient to work with compositions.

The further application of the 2-component equations and the 2-component calculation method of McCabe and Thiele to fractional distillation of the n-component mixture by continuous and batch methods does not appear to be correct. For example, in graph No. 2, page 823, the McCabe and Thiele method is applied to a group of " normal " equilibrium curves. We know that the equilibrium curves of the intermediate components do not actually have this shape. For many of the components the curves cross the 45 degree line and for some the curves take the form of two loops. The intermediate components are the ones that constitute the overlap and the underlap, and the conclusion follows that the deductions resulting from the method used in this portion of the paper cannot be correct. No experimental data are presented in connection with this section of the paper.

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* *J. Inst. Petr. Techn.*, 1933, **19**, 819–834.

In general, the use of a standardized numerical expression to denote degree of fractionation appears to be of doubtful value. If a column is operating to produce an endpoint gasoline as overhead, there will always be some lap in the true-boiling-point curves of the overhead and bottoms. If the endpoint of the gasoline is as desired, this lap is all "underlap" and no "overlap" exists from the commercial standpoint. For this case, the percentage of material in the bottoms which could theoretically be included in the overhead is probably the most satisfactory numerical expression for the degree of fractionation. If some characteristic of the product, other than endpoint, is the criterion for operation, some other expression would probably be the most satisfactory as an indicator of the relative efficiency of the apparatus. If a standardized numerical expression is to be of value, it must be capable of ready translation into terms of commercial loss in order to avoid misleading indications. It is probable that the fundamental information in regard to overlap in terms of our commercial needs will be more valuable than any further numerical expression that can be derived from it.

Mr. F. C. Cutting writes: This paper suggests the use of an overlap coefficient as a basis for comparing the results obtained in different distillation systems. This coefficient is defined by the following terms:—

1. *Overlap.*—The volume of material in the distillate from a specified volume of feed which boils above the cut-point temperature.

2. *Underlap.*—The volume of material in the residue from a specified volume of feed which boils below the cut-point temperature.

3. *Cut-point Temperature.*—The average boiling point of the overlap, underlap or the temperature at which the same percentage overhead is obtainable by standard column distillation. Overlap volume is the overlap coefficient where the specified volume of the feed is such as to give a distillation curve of 1 ml. per 1° C. in the cut-point region of the standard column curve. This specification provides a common base to which observed overlaps can be reduced for comparison.

An interesting derivation is presented in the discussion of equilibrium flash vaporisation, and it may be noted that the conclusion arrived at here checks the practical observations of Obryadchakoff,[1] who concluded that "Fractions which have distillation curves with equal slopes will have the same equilibrium curves." In the paper under discussion, a constant factor for all such vaporisations is accomplished by plotting the distillation curve for such a volume as to give a predetermined constant slope in the cut-point region. Katz and Brown[2] also discuss a similar empirical method for calculating the results of equilibrium flash vaporisation. They

use the 10 per cent. and 70 per cent. points of the distillation curve for determination of its slope. Obryadchakoff uses the 20 per cent. and 80 per cent. points, and in the paper under discussion the cut-point region is used.

It is desired to point out, also, that Katz and Brown recommended corrections to the equilibrium curve if the vaporisation occurs under a pressure other than one atmosphere; this is in reality a correction for different temperature of vaporisation which causes a different relationship between vapour pressures of various components. This point is important in regard to the application of similar equilibrium functions to fractional distillation calculations.

In the comparison of continuous and batch distillation processes, the results obtainable from a given fractionating system are calculated by use of the McCabe and Thiele graph. Equilibrium curves are constructed for selected pairs of components with boiling points equidistant from the cut point on the temperature scale, and the series of curves is explored by the graphical computation method in order to determine the composition of the top and bottom products for a selected reflux ratio.

Three sources of error are inherent in this method of computation :—

1. The components are separated in a column through which there is a temperature gradient. Therefore, an equilibrium curve based on a constant K value (constant temperature) cannot accurately represent the condition under which any pair of components is separating throughout the column.
2. The temperature gradient in a column is a function of the reflux ratio. Hence, even neglecting the common error introduced in the assumption of a constant K value, the comparison of two different reflux ratios on the basis of the same K value for corresponding pairs of components leads to some inaccuracy.
3. The McCabe and Thiele diagram represents accurately the material balance equation of any one component, but usually does not apply to any two components in a mixture of several when these two are treated as a binary mixture. Except in the case of infinite reflux, the operating lines for different pairs of components do not have the same slope, and in many cases these operating lines are not straight, even though the ratio of total reflux to total vapour is constant in each section of the fractionating column.

It is regretted that time is not available in which to illustrate the effect of these three sources of variation on the computations made in the reference paper, and that another example for which

data are available will have to be used instead. This example comprises calculations on the fractionation of a feed of the following assumed composition :—

	Mol. %.		Mol. %.
i-butane	3·3	nonane	9·1
n-butane	4·1	decane	6·9
i-pentane	5·5	undecane	5·5
n-pentane	7·8	dodecane	5·0
i-hexane	4·7	tridecane	4·8
n-hexane	4·1	tetradecane	4·1
n-heptane	15·3	penta decane	3·6
n-octane	12·1	hexadecane	4·1

Several fractionations of this feed were computed by the method of Thiele and Geddes[3] for a 10-plate column (11 plates, including the reboiler) with different reflux ratios to produce in each case an overhead product containing slightly less than 0·1 mols. of tridecane. In each case the feed was vaporised sufficiently to increase the molal quantity of vapour at the feed plate by an amount equal to one-half the feed. The ratio of total vapour to total liquid in each section of the column was assumed to be constant.

The following plate temperatures were found for reflux ratios of 1/2 : 1 and 5 : 1 :—

	1/2 : 1. °F.	5 : 1. °F.
Top Plate No. 1	308·5	313·0
„ „ 2	348·0	386·0
„ „ 3	359·5	411·0
„ „ 4	365·5	420·5
„ „ 5	370·5	428·0
„ „ 6	376·3	434·4
Feed Plate No. 7	386·2	442·0
„ „ 8	450·0	462·0
„ „ 9	468·0	474·0
„ „ 10	483·4	489·0
Reboiler	502·0	508·0

The three items listed above will be discussed on the basis of these data and other information which follows :—

1. Variation in vapour pressure ratios or " K " values. These are shown below for a few combinations of components and for the temperature gradient of the 1/2 : 1 reflux.

	" K "			
Combination.	Plate 1.	Plate 2.	Plate 10.	Reboiler.
C_{11} and C_{13}	3·36	2·99	2·23	2·165
C_{10} and C_{11}	10·51	8·64	5·25	5·01
C_8 and C_{16}	115·3	79·6	31·0	28·3

These combinations are not composed of components boiling exactly equidistant from the cut point, but they serve equally well to indicate the variation in K values that may occur due to the temperature gradient in the column.

2. Variation in temperature gradient with reflux ratio. A comparison of the two temperature gradients listed above shows that whereas the end temperatures differ by only 5–6° F., the intermediate temperatures differ by 50–60° F. Consequently, the average volatility ratio—if such a term may be used—is different for the same combination of components under the different operating conditions. There is no manner in which this divergence can be compensated for in the graphical computation method used in the reference paper. It should be stated, also, that only actual check computations will show whether or not this item has caused any serious error in the computations reported therein.

3. Reflux ratio for a pair of components treated as a binary mixture.

For the case of the 1/2 : 1 reflux ratio, the concentration of undecane and tridecane at each plate are given below as mol. per cents. :—

Plate No.	Undecane.		Tridecane.	
	Liquid.	Vapour.	Liquid.	Vapour.
1	25·6	6·536	1·147	0·087
2	27·84	12·89	2·842	0·441
3	25·13	13·63	5·280	1·006
4	21·54	12·73	8·605	1·818
5	18·24	11·54	12·78	2·926
6	15·24	10·44	17·16	4·319
7	12·10	9·43	19·74	5·778
8	9·08	15·08	26·29	18·45
9	5·59	11·24	30·47	26·78
10	2·88	6·79	30·37	32·11
Reboiler	1·18	2·83	24·45	31·98

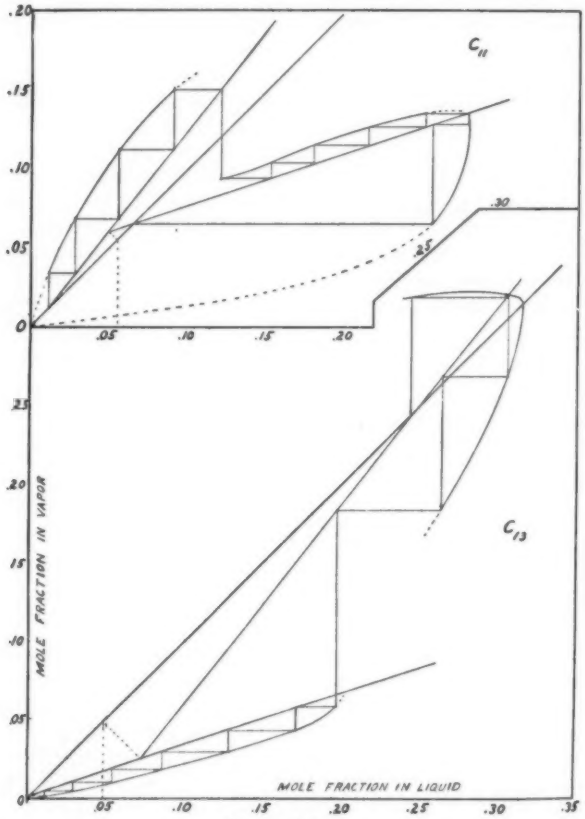
These values plot as shown on graph No. 1. It is evident that these concentrations of each component quite accurately outline a McCabe and Thiele graph in which the operating lines represent the column conditions of total liquid and vapour flow : ratio of reflux to vapour in rectifying section =0·333 ; ratio of vapour to reflux in stripping section =0·786.

If these components are considered to be operating as a binary mixture in the fractionating column, the reflux ratio for these two can be calculated by adding their compositions in the

vapour and liquid at each plate and calculating the ratio on the basis of the known total quantities of vapour and liquid. For example, between plates 1 and 2 the ratio is calculated as follows :—

Total per cent. of C_{11} and C_{13} in reflux liquid = 25·6 / 1·147 =26·747.

Total per cent. of C_{11} and C_{13} in vapour from Pl. 2=12·89 / 0·441 =13·331.



GRAPH NO. 1.

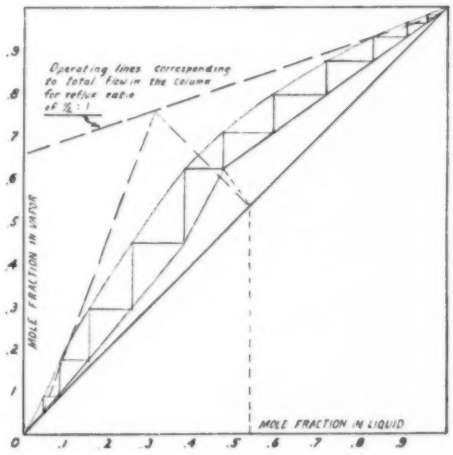
MCCABE AND THIELE GRAPHS FOR C_{11} AND C_{13} IN 10-PLATE FRACTIONATING COLUMN REFLUX RATIO $\frac{1}{5}$: 1.

The vapour quantity is three times the liquid quantity, so the ratio of reflux to vapour for the combination of these two components is :—

$$\frac{26{\cdot}747}{13{\cdot}331 \times 3} = 0{\cdot}668.$$

Similar computations throughout the column give the following ratios for these two components. These ratios are compared with the ratios for total composition.

	Ratio for C_{11} and C_{13}.		Ratio of Total.	
	Reflux : Vapour.	Vapour : Reflux.	Reflux : Vapour.	Vapour : Reflux
Above Plate 1	0·333	—	0·333	—
„ „ 2	0·668	—	0·333	—
„ „ 3	0·700	—	0·333	—
„ „ 4	0·697	—	0·333	—
„ „ 5	0·694	—	0·333	—
„ „ 6	0·702	—	0·333	—
„ „ 7	0·710	—	0·333	—
„ „ 8	—	0·826	—	0·786
„ „ 9	—	0·844	—	0·786
„ „ 10	—	0·848	—	0·786
„ Reboiler	—	0·830	—	0·786



GRAPH NO. 2.

MCCABE AND THIELE GRAPH FOR C_{11} AND C_{13} TREATED AS A BINARY MIXTURE IN 10-PLATE FRACTIONATING COLUMN.

From this comparison, it is evident that the condition applicable to the binary mixture is materially different from the condition of total material flow within the column. This is shown also by graph No. 2, in which the data for undecane and tridecane are plotted, assuming that these components constitute a binary mixture. It is apparent from this plot that the separation between these components actually obtained—in the presence of the other components—is indicated to be impossible if operating lines are drawn in corresponding to the total material flow in the column.

It happens that the boiling points of undecane and tridecane are very nearly equidistant from the cut point (–21° C. and / 18° C. respectively). In order to determine approximately what variation in liquid vapour ratio occurs for different pairs of hypothetical components boiling equidistant from the cut point, the compositions of the liquid on plate 5 and the vapour from plate 6 were plotted as continuous curves. These curves were integrated to determine the composition in terms of hypothetical components of stated narrow boiling range. The liquid vapour ratios for various binary mixtures composed of pairs of these components boiling equidistant from the cut point were computed with results as listed below :—

°F. from Cut Point.	Ratio of Liquid to Vapour between Plates 5 and 6.
10	0·854
20	0·923
40	0·753
60	0·384
70	0·375

Very small concentrations of heavy materials in the vapour made calculations for greater divergences unreliable. However, these figures indicate generally high ratios for pairs of components boiling near the cut point and lower ratios for the pairs with boiling points more distant. Very low ratios must occur for pairs with boiling points still more distant in order to bring the overall average to 0·333.

It appears that there is no possibility of correcting the graphical computation method for such deviations as these.

Consideration of the three items discussed above leads to the conclusion that the results calculated in the reference paper to be obtainable from continuous distillation may be considerably in error. As an indication of the error involved, the overlap coefficient for the example discussed here (1/2 : 1 reflux) is 5·4—a lower coefficient than indicated by the graph opposite page 827 for eleven plates and 1 : 1 reflux. Therefore, the basis for the conclusions

stated at the end of the reference article appears to be of doubtful value.

It is desired to draw attention to conclusion No. 4 : " The more plates a column has, the higher is the reflux ratio necessary to make full use of the plates." It is evident from the example discussed herein that in the continuous fractionation of a long boiling range mixture at a low reflux ratio the reflux ratio for combinations of components boiling near the cut point may be quite high ; such components constitute the major portion of the overlap and underlap volumes and, consequently, the plates are very useful.

Mr. P. Meyer, in reply, writes : Mr. Roelfsema refers to the shape of the equilibrium curves for the intermediate components, and states that these are known not to be " normal." In the author's view, the weight of experimental evidence available suggests that they do not deviate much from a " normal " curve. If Raoult's Law or Brown's Law[1] holds, then a normal curve will be obtained, and the experimental work of Brown and Caine[2] and of Goodliffe[3] indicates that a law of this type is generally applicable. Furthermore, the agreement shown in graph 1 of the reference paper between experimental results and the results calculated, assuming Raoult's (or Brown's) Law, indicates the general validity of a law of this type.

Both correspondents refer to the lack of constancy of the vapour pressure ratio, K.

A large variation in the value of K is associated with :—

(*a*) A large temperature difference between the column-head and the reboiler.

Also :

(*b*) A large difference in the boiling points of the components considered.

Now condition (*a*) is that of fractional distillation, under which components of large boiling-point differences are more or less completely separated into distillate and residue. These components contribute very little to the amount of overlap or underlap, and thus have a negligible effect on the overlap coefficient.

In the case detailed by Mr. Cutting, it is shown that the vapour pressure ratio has a four-fold variation in magnitude for C_3/C_{16}, which boil about 80° C. away from the cut point, and a two-fold variation for C_{10}/C_{14}, boiling about 40° C. away from the cut point.

These pairs of components are practically completely separated, however. Mr. Cutting himself refers to this fact in the fourth from last paragraph of his comments, where he states that : " Very

small concentrations of heavy materials in the vapour made calculations for greater divergences unreliable "—e.g., for divergences greater than 70° F.=39° C. from the cut point.

These components therefore exert a negligible effect on the overlap-underlap volumes, and the wide variation in the value of K is without significance in this connection.

In the case quoted, the overlap-underlap consists mostly of C_{11}/C_{13}, for which the value of K is much more nearly constant, varying from 2·165 up to 3·36. It may be reasoned that the use of an average value for K, where the variation of K is small, does not introduce a serious error, and is sufficiently accurate for approximate work. In this connection Underwood[4] gives a detailed examination of the error involved in assuming K constant at an average value, for distillation at total reflux, and finds that no serious error is introduced.

Mr. Cutting quotes an absolute figure for the overlap coefficient, calculated for a specific case by the Thiele and Geddes method, and compares it with the relative results shown in the reference paper.

The overlap coefficient for the case detailed, of a ten-plate continuous column at a 0·5 : 1·0 reflux ratio, works out at 7·5 by the method in the reference paper, as compared with a value of 5·4 by the Thiele and Geddes method. In the author's view the agreement is within the range of accuracy envisaged by the paper.

Regarding the effect of reflux on a multi-component distillation system, the method of Thiele and Geddes does of course yield precise results for the component distributions. The assumption made in the reference paper on this point is stated on page 824, " that the total overlap is the sum of that obtaining for each twin-component mixture," which is clearly an approximation.

Commenting on conclusion No. 4 of the paper, Mr. Cutting suggests that for the case quoted by him a little reflux goes a long way. If, however, the case were calculated for twenty plates, as well as for ten plates, the comparison would, I think, show that the additional ten plates would do very little work unless the reflux ratio were also increased. In some recent experimental results reported by Goodliffe,[3] it was found that more than half the plates of a twenty-two plate column were ineffective, being operated at too low a reflux ratio.

Regarding the possibility of comparing distillation systems in the manner proposed in the reference paper, Mr. Roelfsema is understood to suggest that any theoretical treatment is only of use if relevant to commercial testing methods. Using, as he proposes, " the percentage of material in the bottoms which could theoreti-

cally be included in the overhead " as a numerical index of fractionation efficiency, the simple case of flash vaporisation shows very variable efficiencies dependent upon local factors such as the boiling range of the mixture, the shape of the distillation curve, the amount of residue formed, and so on ; whereas Mr. Roelfsema agrees in his second paragraph that some basic index for this case, independent of these local variables, may be possible.

Apart from the question of large-scale commercial significance, it is frequently convenient to duplicate large-scale distillation results in the laboratory by, e.g., a small-scale batch distillation. It is well known that cutting to an end-point overhead, for instance, does not necessarily yield a laboratory product comparable with the large-scale product, and some correlation of distillation systems would be advantageous.

The complexity of the subject may render the derivation of a strictly accurate comparative index a matter of some difficulty. Undoubtedly with the progressive application of mathematical methods in chemical engineering and physical chemistry, some modification of the reference paper will be found necessary ; but it would perhaps be premature to assume that no such index is possible. As a parallel in complexity the author suggests the subject of heat transfer by convection ; Langmuir,[5] in 1912, referred to Russell's[6] comments on the number of variables involved and the improbability of correlating them all, and proceeded to show how, in one case at least, that of long thin cylinders, a tenable theory bore practical fruit. Neither Langmuir nor Russell were quite correct ; but it now appears[7] that practically every variable may be taken into account in a reasonably accurate manner.

REFERENCES.

[1] *Ind. Eng. Chem.*, 1932, **25**, 1155.
[2] *Ind. Eng. Chem.*, 1933, **24**, 1373.
[3] *Ind. Eng. Chem.*, 1933, **25**, 289–295.
[1] Sidney Young, " Distillation Principles and Processes," 1922, 80.
[2] Brown and Caine, *Trans. Amer. Inst. Chem. Eng.*, 1928, **21**, 87.
[3] Goodliffe, " The Practical Testing of a Continuous Petroleum Still."
[4] Underwood, *Trans. Inst. Chem. Eng.*, London, 1932, **10**, 120.
[5] Langmuir, *Physical Review*, 1912, **38**, 401.
[6] Russell, *Phil. Mag.*, 1910, **20**, 591.
[7] W. J. King, " The Basic Laws and Data of Heat Transmission," *Mechanical Engineering*, March-August, 1932 ; also C. H. Lander, " Modern Methods of Attacking Heat Transmission Problems," paper read at the Twelfth Annual Corporate Meeting of the *Inst. Chem. Eng.*, London, 1934.

CORRESPONDENCE.

To The Editor.

Sir,—In my "Transformations of Petroleum in Nature" in the March number of the Journal, the following errors were not caught in the manuscript and there was not time to send the proof across the ocean for correction :—

Under the legend of Fig. 1A, under "B" read 275°–300° C., instead of 200°–225° C.; and under "Character of Base" No. 2, read 33·1 instead of 30°; under 3 and 4, 33° A.P.I. or less, instead of 30°. Similar corrections should be made at the top of page 208 and in the legend of Fig. 3.

Houston, Texas, April 11th, 1934. Donald C. Barton.

REVIEW.

Steinkohlenteer. By Dr. Fritz Rosendahl. No. 33 of the Fortschritte der chem. Technologie in Einzeldarstellungen. Edited by Prof. B. Rassow. Dresden and Leipzig: Theodor Steinkopff, 1934. Illustrated. Pp. 199. Price: sewn, 13 RM.; bound, 14 RM.

This is a technical book of limited size, packed with orderly information, carrying out the purpose of the series by bringing together within one pair of covers accounts of the latest advances scattered throughout chemical-technical literature.

Interest is taken primarily in high-temperature tar, of which the formation is traced from the first-formed low-temperature distillates. Considering the high complexity of coal tar, it is remarkable to note (on the basis of work by Weiss and Downs) that less than 10 per cent. of the whole mixture consists of unknown materials :—

Unknown neutral oils (middle and heavy)	1·7	per cent.
„ acenaphthene derivatives	1·0	„
„ fluorene derivatives	1·2	„
„ anthracene derivatives	5·4	„
	9·3	„

A fully documented list follows giving the undoubted constituents of coal tar, which are then reproduced in the form of a genealogical table. The colloidal nature of tar, and the separation of the latter into its constituents by differential solvents and by distillation, are closely described, and, in accord with the whole attitude of the book, are kept in close association with the industrial aspect and significant patents.

The working-up of the various fractions takes up about a third of the book, and is full of interest. Then follow the latest details concerning tarred roofing felt; also of road tar and associated aggregate for surfacing, and the final mixtures. Together with these are described the less usual forms of tar—filled tar and tar emulsions, "cold tar," and tar-bitumen mixtures.

Name and subject indexes, and a few pages of advertisements, complete a useful and interesting volume. Percy E. Spielmann.